8-2009

The effect of bio-based materials on quality and shelf life of celery

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THE EFFECT OF BIO-BASED FILMS ON QUALITY AND SHELF LIFE OF FRESH CELERY

A thesis

Presented to

The Graduate School

of Clemson University

In partial Fulfillment

of the Requirements for the Degree

Master of Science

Packaging Science

By

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August 2009

Accepted by:

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ABSTRACT

Bio-based materials have garnered increasing interest as food packaging materials due to their raw materials derived from renewable resources such as corn starch, cellulose, and sugar beets. One important drawback is that they can exhibit poor mechanical performance compared to non-bio based materials and their effect on quality and shelf life of some produce is unknown.

This research studied the effect of three bio-based materials on quality and shelf life of fresh celery. Materials include non-perforated biopolymer films and perforated low density polyethylene (LDPE) film. The variable materials were: Polylactic Acid (PLA), a versatile biodegradable aliphatic polyester derived from 100% renewable resources, Ecoflex, a biodegradable aliphatic-aromatic copolyester, and Mater-Bi, a bio-plastic derived mainly from natural renewable resources such as corn, wheat, and potato starch. The control was perforated LDPE.

The objective of this research was to determine if biopolymer films could be used to package fresh celery and if they were comparable to the currently used packaging material (LDPE).

Whole (uncut) fresh celery stalks packaged into sleeves made from the above materials were subjected to refrigeration conditions (5°C & 95%RH) and bi-monthly analysis for 3 months. The analysis included appearance, weight loss, microbiology, sensory, texture, and petiole color. Material or film analysis included WVTR, OTR, Tensile Strength, and Elongation.

The results showed that product quality attributes did not significantly differ between materials. Consequently, celery could be packaged in any of the materials utilized in the study

and maintain natural quality over time as well as naturally deteriorate over time. With respect to weight loss, celery packaged in all bio-based materials experienced decreases in weight. Celery packaged in Mater-Bi material had the least weight loss at end of study. Results also demonstrated material deterioration occurring in both Ecoflex and PLA materials based on tensile and break elongation results under the high humidity conditions of the present study. Both materials also displayed the highest increases and fluctuation in permeation rates. While Ecoflex and PLA materials were suitable materials with respect to product quality attributes, their mechanical properties would need to be improved to match the performance of Mater-Bi. Mater-Bi was superior to other bio-based materials with regard to mechanical performance and therefore would be recommended for storage of fresh celery over other bio-based materials.

DEDICATION

I dedicate this work to my parents and all family members in Nigeria who have always advocated higher education and a thirst for continuous learning.

ACKNOWLEDGEMENTS

I sincerely offer my heartfelt gratitude to my thesis advisor, Dr. Kay Cooksey, for her patience and guidance throughout my research study. A special appreciation to my committee members Dr. Duncan Darby, and Dr. Robert Kimmel who have been patient and willing to offer a fresh start when I made mistakes and gone astray. I also extend appreciation to my sensory coordinator, Mrs. Halpin for her assistance in coordinating sensory analysis and soliciting panelists. Thanks to Dr. Whiteside for organizing the distribution and arrival of a storage cooler and for his challenging style that assisted in enriching the body of this work.

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CHAPTER 1

INTRODUCTION

Most plastic packaging applications utilize fossil fuel based polymers which are highly non-biodegradable and difficult to recycle or reuse due to blends of materials, impurities, and complex composites. The disposal of packaging waste serving as a main part of municipal solid waste and the rising cost of petroleum based materials has revived concerns prompting actions to significantly reduce amount of packaging waste (Bastioli, Catia, 2005). Consequently, the advent of biodegradable packaging materials from renewable resources may potentially play a role in contributing to sustainable development by having a reduced environmental impact (Bastioli, Catia, 2005). For instance, bio-based materials can serve as mulch films that protect soil and plants against weed growth, raise yields and elevate soil temperatures, then can be disposed by plowing into the soil.

Although modern plastics such as polyethylene (PE), polypropylene (PP), polystyrene (PS), and polyethylene terephthalate (PET) are strong, inexpensive, and durable, they are resistant to biological degradation due to inability of their carbon components to be broken down by microorganisms (Bastioli, Catia, 2005). Additionally, the hydrophobic nature of synthetic plastics, coupled with their low surface area and high molecular weight, further inhibits enzyme activity and compounds their resistance to microbial attack (Bastioli, Catia, 2005).

The advent of biodegradable polymers that are readily manufactured, possess good mechanical properties, and are derived from renewable resources, can compete strongly in the market place alongside petroleum based counterparts. If biopolymers are to replace some of the synthetic polymers, more research is needed to determine what effects they will have on the quality and shelf life of foods.

CHAPTER 2

LITERATURE REVIEW

Celery Characteristics

Variety

The Celery plant species is called *Apium graveolens*. It yields two important vegetables which are celery and celeriac. Celery is a biennial crop that later becomes an annual crop when early or warm weather causes development of a flowering stem. Biennial crops require two years to complete their life cycle. During the second year, they begin to flower and produce other botanical structural components such as flowering, seeds, etc, that are indicative of a mature crop (APIACEAE- The Celery Family).

Celery belongs to the family of *Apiaceae*. Over 2500 members of the *Apiaceae* family are found worldwide at predominantly temperate regions. Other notable vegetables and herbs found in this family include carrots, parsnip, fennel, and ornamental garden plants such as *Eryngium*, *Astrantia*, and *Aciphylla*. Characteristics of the plant family *Apiaceae* are indicated in Figure 1.1.

Leaves, Stem and Root

Apiaceae family members are soft-stemmed annuals, biennials, or perennials (APIACEAE- The Celery Family). At the leaf joints, stems can be hollow or ribbed as celery, while the leaves are divided.

Figure 1.1 *Apium graveolens* of the Wild Celery Variety
(USDA Plants)

Flowers

Flower stalks grow from the leaf axil. The axil is the angle between the upper side of a leaf or stem and the stem or branch that supports it (APIACEAE- The Celery Family). As the flowers grow, they form into clusters in an umbrella shape. Outer flowers may bloom first and can appear in colors of white, cream or yellow. The appearance of members in the *Apiaceae* family grow to be relatively lanky and appear gangling in similar fashion to an asparagus.

Seeds

The seeds of the *Apiaceae* family are comprised of different shapes and sizes ranging from winged shaped to spiny. Located behind the petals, seed capsules consist of two parts, each having a single seed.

Factors Affecting Shelf Life of Celery

Overall quality of a wide assortment of produce such as broccoli, lettuce, and celery is determined by quality factors which include weight loss, microbial spoilage, appearance (visual characteristics), and sensory qualities (Kilcast & Subramaniam, 2000). Consequently, those quality criteria are important determinants of product shelf life. Quality criteria also depend on specific commodities and the condition in which the commodity is sold. For instance, quality criteria for produce sold fresh will differ from that sold minimally processed. For wholesome uncut non- processed celery sticks, the key factors and criteria that affect shelf life and quality are grouped into the categories of appearance, texture, and flavor/aroma. Initially, signs of quality decay and decreasing shelf life for celery are the loss of green color and the onset of pithiness (Gomez & Artes, 2005). Pithiness is characterized by the appearance of whitish regions and air spaces within celery tissues and leads to reduced tissue density (Luo, Suslow, & Cantwell, 2002). Pithiness is identified as a major source of quality loss and decreased shelf-life in celery (Saltveit & Mangrich, 1996). Excessive development of pithiness can be retarded by storage at low enough temperatures (0°C, 32°F) which prolongs shelf life of the celery.

Shelf life of vegetable products alike can vary depending on storage conditions. A vegetable product such as celery will rapidly degrade if storage conditions are incapable of maintaining and or retaining inherent moistness or water activity. Since celery contains 94.7% water (Bartz & Brecht, 2003), the shelf life of wholesome uncut celery at optimum storage conditions (0°C, 32°F & > 95%RH) is 5-7 weeks (Hardenburg, Watada, & Wang, 1986). Otherwise, conventional refrigeration conditions limits shelf life to approximately three weeks.

Celery shelf life, quality, and storage can be limited by any defect in appearance. Appearance is a critical feature in consumer decision making regarding purchase of fresh celery. Fresh celery is expected to exhibit zero defects in appearance while also displaying evenness in color, shape, and size. Visual celery quality also includes lighter green areas underneath the inner surface of celery sticks and heavier green areas on the outer external areas of celery sticks. Conversely, the yellowing of a green vegetable, such as celery, is an indication of accelerated maturation and thus signals the end of the shelf life. The other critical appearance characteristics that affect shelf life include wilting, skin wrinkling, and shriveling.

Just as appearance characteristics affect shelf life of celery, textural properties also provide an indication of celery quality. Good quality celery is associated with crisp and firm textural attributes. In contrast, developments of tough textural attributes are undesirable and closely associated with product maturation. Textural attributes can also be visually identified via appearance indications of wilting and shriveling. While textural and appearance attributes can be vividly and physically observed, flavor/aroma attributes are another key category of celery that affects quality and shelf life. Flavor can not be easily evaluated by consumers prior to purchase of a vegetable product such as celery. Nevertheless, vital celery flavor characteristics include bitter, aromatic, astringent, and salty. Unlike flavor, aroma can be evaluated before purchase by consumer. For fresh produce such as celery, aroma is a pivotal quality component that consumers utilize in determining freshness and appeal of celery sticks before purchase. Unpleasant odors that may indicate putridity can develop due to mechanical damage to cell tissue during distribution which renders the produce highly vulnerable to microbial contamination. Microbial agents such as *Pseudomonas fluorescens* and *P. marginalis* have been identified as decay and soft rot causative agents in fresh cut celery (Robbs, Bartz, Mcfie, & Hodge, 1996).

Such microbiological agents are responsible for unpleasant aromas which can render a product unmarketable despite other quality factors being desirable. Consequently, aroma is an important factor in the quality, storage, and shelf life of celery. (Selke, Cutler, & Hernandez, 2004)

BASIC MATERIAL PROPERTIES

Low Density Polyethylene

Low Density Polyethylene (LDPE) is a thermoplastic polymer made from petrochemicals and is the most widely used packaging plastic for a variety of manufacturing applications including plastic bags, tubing and food packaging containers. A thermoplastic can be defined as a polymer that can be shaped multiple times at reasonably low temperatures (Kimmel 2006) that can be heated, formed and cooled into a new shape. Belonging to the polyethylene resin family, LDPE is classified as a branched polymer due to a high degree of short and long chain branching (Selke, Cutler, & Hernandez, 2004).

Shortly after its introduction in the 1950's, polyethylene became a common material used in film production, and container closures (Selke, Cutler, & Hernandez, 2004). Additionally, mechanical properties such as strength, toughness, heat sealing properties, etc, have considerably improved since its introduction as a premier packaging material. LDPE possesses a branched structure similar to other members in the family of branched polyethylenes which include homopolymers and copolymers of ethylene that are non-linear, thermoplastic, and partially crystalline. Homopolymers are polymers formed from only one type of monomer while copolymers are polymers composed of two or more different types of monomers (Selke, Cutler,

& Hernandez, 2004). These polymers are manufactured under high pressure and temperature parameters via free radical polymerization processes. The manufacture of ethylene via such polymerization conditions produces a branched polymer composed of a blend of large molecules with different backbone lengths, variation of side chain lengths, and various degrees of side-chain branching (Selke, Cutler, & Hernandez, 2004).

Chain branching in LDPE offers a variety of attractive mechanical properties which include clarity, flexibility and heat sealability. In addition to such properties, LDPE is adaptable to a variety of processing means such as blow molding, injection molding, and film casting. Film casting is particularly relevant in the packaging industry because LDPE films serve as the largest volume of LDPE produced, as well as the largest use for packaging applications (Selke, Cutler, & Hernandez, 2004). LDPE production into films comprises of bags for food, industrial liners, vapor barriers, agricultural films, and shrink/stretch films. Serving as the most widely used plastic in packaging, LDPE can be used alone or in combination with other polymers in the polyethylene family. Some other important features of LDPE include: (Selke, Cutler, & Hernandez, 2004)

- Excellent flexibility
- Good impact resistance
- Fair machinability
- Good oil resistance
- Fair chemical resistance
- Good heat sealing characteristics
- Low cost ($1.60/kg)

BIO-BASED MATERIALS

Bio-based materials are derived from renewable sources such as corn starch, cellulose and polysaccharides. They have garnered increasing interest as materials that begin to address the emerging challenge for the 21st century polymer manufacturing for a wide array of applications (Bastioli, Catia, 2005).

The selection of bio-based materials for a range of packaging applications has been based on the evaluation of polymer properties as well as recyclability and sustainability of such polymers. Materials utilized in this study such as Polylactic Acid (PLA) polymer have garnered increasing attention in recent years as consumers demand use of bio-friendly packaging materials (Auras, Singh, & Singh, 2005). Additionally, decreasing availability of landfills (mainly in Europe) triggered by lingering issues surrounding degradation of municipal solid waste have also enhanced consumer interest in packaging materials that are capable of biodegrading.

Biodegradation involves the actions of a microorganism's extracellular enzymes in breaking down a polymer (by attacking ends of large molecules) into products or fragments that are small enough to be assimilated (Bastioli, Catia, 2005). Polymer fragments must be broken down into small enough chain lengths for degradation to occur because enzymes are incapable of digesting larger macromolecules. In biodegradation of polymers, the first step is a chain cleavage step involving the conversion of a long polymer chain into smaller oligomeric fragments. Secondly, small size oligomeric polymer fragments are converted into biomass such as minerals, salts, CO_2 and methane (Bastioli, Catia, 2005)

Specific Bio-based Materials:

Mater-Bi

Mater-Bi (manufactured by Novamont) is made of extracted corn starch coupled with the integration of various synthetic polymers such as poly- ϵ- caprolactone and polyvinyl alcohol to increase flexibility and resistance to moisture. Mater-Bi is a bio-plastic material with similar properties (i.e., flexibility and hydrophobicity) to conventional plastics. Due to the use of natural materials as the constituents, products made from Mater-Bi biodegrade to carbon dioxide, water and organic matter with no toxic residue when subjected to an environment containing bacteria (Plastral-Bioplastics).

Suitable for injection molding and sheet film applications, Mater-Bi conforms to the European EN 13432-2000 requirement for packaging recoverable through composting and biodegradation (Plastral-Bioplastics). Various packaging materials for a variety of applications made from conventional plastics can also be made from Mater-Bi in order to offer various environmental advantages. These advantages include low environmental impact, biodegradability, and non toxicity to marine and terrestrial wildlife. The variety of applications that utilize Mater-Bi include (Plastral-Bioplastics):

- Agricultural mulching films
- Carrier bags
- Liners for separate organic waste collection
- Personal care products
- Catering products (cutlery, plates and cups)
- Food packaging

- Pet products
- Expanded packaging products
- Produce packaging (storage and distribution)

In the processing of starch and similar renewable raw materials to a bio-plastic film, a limited amount of water is utilized coupled with the maintenance of a confined volume throughout entire process (Plastral-Bioplastics). After heating in presence of water for a certain time period, a homogeneous blend is produced that has the ability to be machinable in injection molding equipments and extruders. Typical, finished products possess the ability to be (Plastral-Bioplastics):

- used like conventional plastic materials
- colored with natural pigments
- heat laminated to paper, cardboard, cotton and other natural fibers
- sterilized by gamma rays
- glued with solvents and water-based adhesives

Polylactic Acid

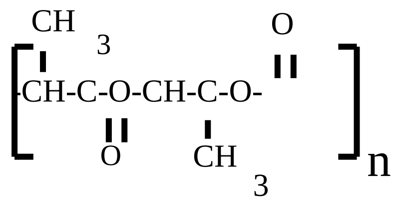

Figure 1.2: PLA Structure (Dugan)

Polylactic acid (PLA), produced by NatureWorks LLC, is a biodegradable aliphatic polyester derived from 100% renewable resources such as corn and sugar beets (Drumright, Gruber, & Henton). Since the lactides from which PLA is ultimately produced can be derived via the fermentation of agricultural by-products, PLA is considered a sustainable alternative to petrochemical derived products (Drumright, Gruber, & Henton). PLA has a wide range of applications ranging from food packaging applications (microwavable trays) to biomedical applications (drug delivery equipment). The biodegradable characteristic of PLA enables it to be utilized in the development of bio-plastic packaging bags such as compost bags and a variety of loose fill packaging utilized in produce packaging. Although PLA is more costly than its petroleum based counterparts, the price has been dropping as production has increased.

The formation of Cargill Dow LLC in 1997 brought two large companies together to focus on the production and marketing of PLA with a goal of reducing production costs and manufacturing PLA in large volumes (Drumright, Gruber, & Henton). PLA can be manufactured by both direct condensation of lactic acid and by the ring opening polymerization of the cyclic lactide dimer. The ultimate molecular weight achieved by the ring opening polymerization approach is generally limited due to difficulties in removing trace amounts of water in the late stages of polymerization. Immense research has been directed towards the ring opening polymerization even through Mitsui Toatsu Chemicals has patented an azeotropic distillation process utilizing a high boiling solvent to drive the removal of water in the direct esterification process in order to obtain high molecular weight PLA (Drumright, Gruber, & Henton).

PLA is suitable for a variety of applications due to an assortment of qualities such as crease retention, crimp properties, excellent grease and oil resistance, easy low-temperature heat sealability, and good barrier to flavors and aromas. Additionally, PLA is also suitable for various mechanical processing applications which include sheet extrusion, film blowing, and fiber spinning.

Due to its degradation capability, PLA is also suitable for environmental applications such as agricultural mulch films and bags in addition to clear food containers and beverage bottles. Such broad application has enabled PLA to be introduced into fresh-cut produce packaging at Sam’s Club and Wal-Mart Super Centers. Since 2005, clear, thermoformed PLA packaging containing fresh cut fruit, herbs, strawberries, and Brussels sprouts has surfaced in grocery stores nationwide (NatureWorks-Sam's Club, 2005). Due to such introduction into the fresh produce chain, there could be potential for savings (gasoline and money) gained from use of a bio-based plastic made from 100% renewable natural resource rather than petroleum based plastic packaging.

Eco-Flex

Ecoflex, produced by Badische Anilin-und Soda-Fabrik (BASF), is an aliphatic aromatic copolyester that can be broken down by an array of common microorganisms in both soil and compost (Mariniello, et al., 2007). Specifically, Ecoflex is an aliphatic aromatic copolyester made of modular units including 1, 4-butanediol, adipic acid, and terephthalic acid (Mariniello, et al., 2007). Novamont and BASF produce such materials by adjusting components and adding special additives such as poly- ϵ- caprolactone and polyvinyl alcohol.

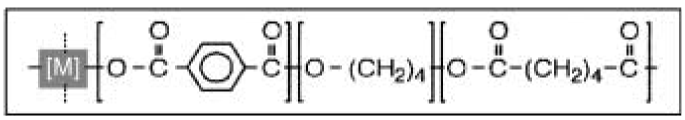

Figure 1.3: Chemical profile of Ecoflex
(Motonori, Witt, Skupin, Beimborn, & Muller)

With modern culture emphasizing renewable raw materials, materials such as Ecoflex have a potential to replace conventional plastics in certain applications where dependence on fossil fuels can be reduced. Below is a list of applications suitable for Ecoflex (Motonori, Witt, Skupin, Beimborn, & Muller):

- Compost bags
- Packaging films
- Horticultural films
- Agricultural films
- Films for household applications

The properties of Ecoflex are designed to meet the requirements of processability, utilization properties, and biodegradability (Motonori, Witt, Skupin, Beimborn, & Muller). Such requirements are achieved by the synthesis of tailor made molecular structures obtained through modular units by which the copolyester units (1, 4-butanediol and dicarbonic acids, adipic acid and terephthalic acid) are linked. The modular system involves the incorporation of hydrophilic components of monomers with branching, thereby leading to chain lengthening and an increase in the molecular weight to yield tailor made products with different material properties (Motonori, Witt, Skupin, Beimborn, & Muller).

Mechanical properties of Ecoflex are reported to be similar to those of Low Density Polyethylene (LDPE) (Motonori, Witt, Skupin, Beimborn, & Muller). Films are tear resistant, flexible, and resilient to water and fluctuations in humidity. They are breathable due to their moderate water vapor permeability (Motonori, Witt, Skupin, Beimborn, & Muller). Ecoflex can be processed via conventional blown film lines for LDPE. The excellent draw down ability of Ecoflex lends some appealing applications in thin film segment with the attainment of 10 µm films. Special handling and pre-drying usually associated with thermoplastic polyesters are not necessary for Ecoflex, thereby providing an additional advantage for the converter. Production of pigmented resins, adjustment of water vapor barrier, antiblock slip properties, and transparency have been developed for extruded film applications to fulfill customer needs (Motonori, Witt, Skupin, Beimborn, & Muller).

Special additives that improve properties allow Ecoflex to be used in applications such as compost bags, films for the agricultural sector, household films, lamination applications, and coating materials for starch based products (Motonori, Witt, Skupin, Beimborn, & Muller). Via integration of special additives and optimizing processing conditions, transparent films can be obtained using blown film process. Such films can then be used for the wrapping of vegetables and fruits. Other applications include the following (Motonori, Witt, Skupin, Beimborn, & Muller):

Compost bags for organic waste

Kitchen waste can be gathered in an Ecoflex bag and composted. The requirements for compost bags include wet strength of the film, time in which it remains stable to the organic waste and problem free processing in a compost facility.

Mulch Films

Mulch films made from Ecoflex utilize the biodegradability characteristic. After harvest, mulch films can be plowed together with the plant residue into the soil, where they fully degrade.

Laminated Materials

Ecoflex as part of a biodegradable laminate offers an advantage of problem free disposal by composting. Lamination is used when there is a necessity for high wet strength and fat resistance. Items of particular interest and relevance include packaging materials soiled with food residues such as paper wraps, paper/plastic cups, and boxes and containers for frozen foods.

MECHANICAL PROPERTIES

The mechanical behavior of a polymer can be evaluated by its stress-strain characteristics under tensile deformation (Selke, Cutler, & Hernandez, 2004). While stress is measured in force per unit area and expressed in psi, strain is the dimensionless fractional length increase (Selke, Cutler, & Hernandez, 2004). Illustrated in Figure 1.4 is a typical stress strain curve (figure 2) depicting the necking and drawing regions (Roylance, 2001).

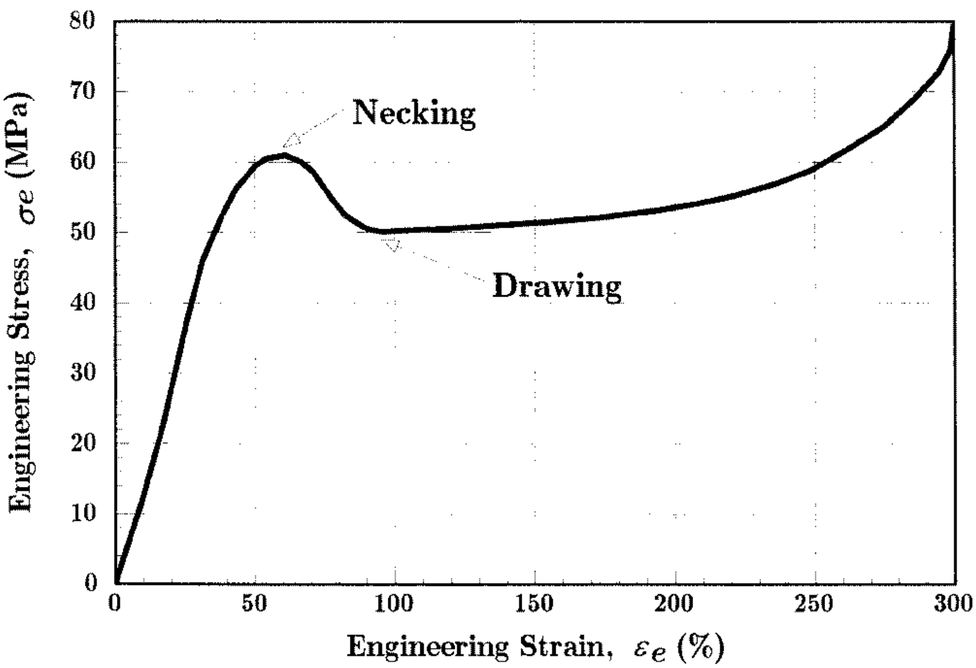

Figure 1.4: Stress Strain Curve**.** Necking or necking-in refers to the tendency of a material's width to decrease during stretching (Selke, Cutler, & Hernandez, 2004) while drawing is the propagation of the necking region until spanning entire gage length of the specimen (Roylance, 2001).

Tensile Properties

Tensile tests are performed to measure mechanical quality of materials during evaluation and development for a variety of applications (Davis, 2004). Tensile properties such as break strength, yield stress, and break elongation can be measured via deformation of a sample while monitoring stress and deformation until the sample breaks (Anker, 1996).

Break strength is the tensile load or force required to break a film (Instron: Materials Testing).

Yield stress is the stress level of highly ductile materials at which large strains take place without further increase in stress (Davis, 2004).

Break elongation is the elongation of a specimen to the break point (Instron: Materials Testing)

Stress-Strain Definition

Engineering stress or nominal stress is defined as $s = F/A_0$, where F is the tensile force and A_0 is the initial cross-sectional area of the gauge section, while Engineering strain or nominal strain, e, is defined as $e = \Delta L/L_0$, where ΔL is the change in gage length ($L – L_o$) and L_o is the initial gauge length (Davis, 2004).

Standard Testing Method

The test method for evaluating tensile properties of thin plastic sheets is identified under ASTM method D-882. ASTM D-882 describes a static weighing test method in which a specimen is placed between two grips that are separated at a constant rate while the applied force and amount of grip separation are recorded (ASTM, 1991).

PERMEATION

Some packaging materials are designed to contain a barrier to permeants such as oxygen and water vapor in order to maintain quality and prolong shelf life of food stuffs such as cereals, granola bars and the like. Conversely, vegetable products such as celery require a barrier that can allow permeation of such permeants as oxygen and water vapor in order to maintain aerobic respiration and adequate cell turgor (turgor refers to the ability of the cell to hold water and provides the plant with a firm texture) necessary to avoid product wilting or shriveling. Permeation is the movement of gases, vapors, or liquids across a homogeneous packaging material (Selke, Cutler, & Hernandez, 2004). Permeation measurements for both oxygen and water vapor exist under ASTM standard methods

Oxygen Permeability Measurement System

Oxygen permeability rate can be measured according to ASTM D 3985 with oxygen permeability testing equipment. During operation, a partial pressure difference across a film with respect to test gases is created without a difference in total pressure. This difference is maintained by sweeping one side of film with a test gas while maintaining an inert gas on the opposite side in order to diffuse the test gas (Robertson, 1993). The use of a Mocon OxTran equipment (Modern Controls, Inc, Minneapolis Minn.) is utilized in measuring oxygen permeability. Mocon OxTran has the capability to also measure the permeability of bottles, pouches, tubes, etc, as well as thin sheet of films (Caner, 1997). The OxTran measuring equipment consists of two chambers of a measuring cell between which the test film is placed. A gas stream of known oxygen partial pressure flows through one chamber while oxygen free carrier gas is passed though the other chamber to a Coulometric detector (Robertson, 1993). A maximum of two test specimens can be installed per testing session. The OxTran operates in conjunction with a computer connected to a disk drive, monitor and printer. After performing the specified testing procedure, a display of the resultant transmission rates for each tested specimen will be displayed in the computer (Caner, 1997).

Water Vapor Permeability Measurement System

One method for determining water vapor permeability (WVPR) is the gravimetric method (ASTM, 1990). The gravimetric method involves sealing a test film in a cup partially filled with water or saturated salt solution thereby providing an air gap beneath the film (Anker, 1996). After sealing a film specimen on the mouth of cup, the assembly is weighed and afterwards held in an environmental chamber under a set temperature and relative humidity (Caner, 1997).

Water vapor flux through a tested film is provided by the partial water vapor pressure difference from the lower side of film to top side of film. Additionally, weight change of the cup, recorded by weighing at scheduled intervals represents WVP from the cup through the film and into the chamber (Caner, 1997). Weight loss is also plotted over time and monitored because when steady state is achieved, the plot is a straight line. To obtain WVPR of tested film, the slope of straight line is divided by exposed film area (ASTM, 1990).

WVTR= slope (g/hr)/area (m^2) × 24hr/day = gram/m^2 day (Robertson, 1993)

WVPR (Water Vapor Permeation Rate) = WVTR × film thickness

Water Vapor Permeability Measurement System: Permatran-6000

A more automated method that utilizes The Permatron-6000 (Modern Controls, Inc. Mocon, Minneapolis, MN) can also measure WVPR of plastic films and permeable barriers alike. Test cells consist of two sections separated by material to be tested. While the lower section contains a fixed amount of moisture, the upper section is connected to a humidity sensor that is able to determine moisture content in dry purge air (Robertson, 1993). Meanwhile, the relative humidity of air surrounding the humidity sensor is elevated via the movement of water vapor through the film, enabling the recording of time for a given rise in relative humidity (Robertson, 1993).

SHELF LIFE OF FOOD PRODUCTS

Shelf life is a critical feature of any food product that conveys the quality and safety of food over time to a consumer before purchase. In general, shelf life of a food product is the time period, from point of purchase till point of consumption, in which a product is microbiologically safe and maintains quality and nutritional attributes (Kilcast & Subramaniam, 2000). Consequently, shelf life is extremely critical to both food processors and consumers. Additionally, food safety and quality attributes are the two major components of an acceptable shelf life. The factors that influence microbiological safety and quality in food are linked. Therefore, in evaluating the shelf life of a food product, those quality attributes that are satisfactory to consumers and essential to consumer acceptance are vital in assessing shelf life of a product.

In assessing the shelf life of food products, the deterioration mechanism of identified quality and safety characteristics must be understood. The end of shelf life is determined by food safety and quality characteristics. Accordingly, during shelf life testing, mechanisms such as moisture gain or loss, flavor/odor migration, microbiological proliferation and sensory changes, can be monitored and used as practical basis to explain, test, and measure product shelf life (Kilcast & Subramaniam, 2000). The general methods for shelf life testing involve sensory analysis, instrumental analysis, chemical measurements and microbiological measurements (Kilcast & Subramaniam, 2000). Each measurement is critical in offering information about the stability of a food product over time. Sensory analysis systematically assesses the changes in eating quality as a function of storage conditions and other environmental conditions for which a product may be subjected. Living subjects were used as participants for sensory analysis in this study. Instrumental analyses are valuable in augmenting and complementing sensory data. Instruments such as texture analyzers and the colorimeters for measuring texture and color properties are extremely relevant especially when striking parallels to sensory data and product attributes over time. Additionally, reactions occurring in foods during storage that are undetectable via sensory and instrumental analyses can be detected through chemical analysis (Kilcast & Subramaniam, 2000). Chemical analysis can be used to identify and measure chemical reactions occurring in food during storage and to validate or explain the degradation mechanism that lead to quality deterioration of a product (Kilcast & Subramaniam, 2000).

Different chemical reactions occur concurrently during storage. Therefore, the major reactions inducing quality changes such as rancidity in a product must be measured during shelf life testing (Steele, 2004).

Another critical quality change that must be addressed is the microbiological stability of a product. The two important aspects to be considered in determining the microbiological stability of a product are microbial growth, which leads to the spoilage of a food product, and the growth of microbial pathogens that affect product safety (Kilcast & Subramaniam, 2000). To identify and enumerate such microbial agents responsible for compromising both product safety and quality, time until spoilage can be determined by storing product at appropriate storage conditions and measuring microbial load at staged intervals (Kilcast & Subramaniam, 2000). Once a predetermined level of microbial count has been achieved, the end of shelf life is declared. Intrinsic product factors such as water activity and pH as well as extrinsic factors such as storage temperature and time can be used to predict shelf life of products.

Shelf life prediction of foods has been developed to meet the need of new products into retail outlets with minimum delay (Steele, 2004). While posing a challenge for the introduction of new longer shelf life products, knowledge of storage characteristics over intended shelf life period is critical in predicting shelf life and avoiding problems of unacceptable delays into the marketplace. Consequently, accelerated shelf life testing is utilized to avoid such problems once there is a valid relationship between storage characteristics under ambient storage conditions and the storage characteristics under accelerated condition. The principle behind accelerated shelf life testing correlates a change in storage condition to an acceleration of the chemical or physical processes that lead to deterioration (Kilcast & Subramaniam, 2000). Additionally, a predictive shelf life relationship related to ambient conditions can be defined. The basis behind such principle is the assumption that the deteriorative processes limiting shelf life remains the same under both ambient and accelerated conditions. Nevertheless, if the above assumption is not correct and another deteriorative process dominates at the accelerated conditions, then a valid relationship between ambient and accelerated conditions is not attainable (Kilcast & Subramaniam, 2000). Some possible deteriorative processes that can dominate include denaturation of proteins, increased water activity, crystallization of amorphous carbohydrates and increased water activity (Steele, 2004).

SHELF LIFE ASSESSMENT OF FRESH PRODUCE

For fresh produce, shelf life is defined as the time period within which the product retains acceptable quality (with respect to appearance, texture, and flavor/aroma) for sale to the consumer (Kilcast & Subramaniam, 2000). Shelf life knowledge of produce is critical to individuals who manage fresh produce supply chains from the grower to the retailer. Certain produce (out of season, highly perishable or short shelf life) may require rapid transportation via air shipment rather than by land or sea. Conversely, others with a longer shelf life can be stored and released according to market demand. Consequently, shelf life measurement of an assortment of fresh produce must be conducted in order to recognize and be forewarned of quality deterioration that may occur during shipment (Kilcast & Subramaniam, 2000).

In shelf life measurement of fresh produce, product samples are removed from the packing line and placed in shelf life storage rooms at a temperature that reflects retail conditions. At those conditions, shelf life tests are conducted at predetermined intervals that assess appearance, texture, flavor/aroma, and microbiological proliferation (Kilcast & Subramaniam, 2000). Appearance is analyzed for uniformity in size, shape and color. Visual quality and acceptable appearance include absence of defects in shape and skin of products (Lamikanra, Imam, & Ukuku, 2005). Visually, an indication of textural quality can also be assessed. For instance wilting and shriveling are appearance indicators but are also indicators of deteriorating textural quality. Crispness and firmness are desirable traits in vegetable crops such as celery. Such traits are easily detected via sensory or organoleptic studies that also assess flavor and aroma. Sensory analysis over predetermined intervals assesses key taste components in fresh produce such as sweetness, acidity, or astringency. Additionally, they also evaluate key textural attributes of crispness and firmness over time as an indication of deteriorating quality. Texture analyzers provide instrumental analysis and solid data over time that can support or complement prior texture analysis conducted via a sensory method.

Finally, microbiological tests will assess the effect of microorganisms on quality and safety of products. Fresh produce are not considered high risk products with regard to food safety because they normally become completely undesirable for consumption prior to complete proliferation by hazardous microorganisms (Kilcast & Subramaniam, 2000).

In addition to real time shelf life testing, Accelerated Shelf Life Testing (ASLT) may be conducted at elevated temperatures to reveal any possible development of pathogenic rots. Subsequently, produce will be subjected to quality assessment (outlined previously) and changes over a predetermined time period for a particular product. Due to different quality and shelf life requirements by individual retailers, samples are assessed from each separate product line. These shelf life tests are designed to raise awareness of potential quality problems that will prompt action to address the issue. Such testing may reveal patterns in quality which can be used in decisions such as when to change the supply source (Kilcast & Subramaniam, 2000). While shelf life predictions can be critical in produce supply chains especially when there are time constraints, accurate shelf life prediction for fresh produce is not feasible due to inherent variability in all quality attributes of fruits and vegetables that are used to determine shelf life (Kilcast & Subramaniam, 2000).

SENSORY ANALYSIS OF FRESH PRODUCE

In measuring shelf life, the underlying assumption about product sensory attributes and eating quality is that a certain level of deterioration occurs over time. Consequently, measurement of changes in eating quality and sensory attributes requires sensory evaluation techniques. Sensory evaluation techniques sometimes involve trained panelists issuing quantitative quality ranks on various quality attributes. Alternatively, untrained panelists can also assess quality attributes on general categories such as overall liking or preference surrounding a sensory quality profile (Kilcast & Subramaniam, 2000). Before sensory analysis, microbiological testing must be completed to assure the safety of individual panelists. There should be an identified benchmark at which bacterial levels must not exceed before conducting sensory analysis. For instance, for minimally fresh processed (immersion in NaOCL water solution) celery sticks, the maximum aerobic bacteria limit is 10^7 CFU g^{-1} (Colony Forming Units) (Gomez & Artes, 2005). If microbiological analysis exceeds 10^7 CFU g^{-1} prior to a scheduled sensory evaluation, considerations should be made for sensory evaluation to be restricted to appearance and odor evaluation.

Sensory evaluation is most effective when a selected panel is utilized in conducting sensory evaluation of a product. Panelists can be trained to detect, identify and assess key quality attributes that are vital to product quality. Such panelists, also referred to as human subjects are generally recruited and screened (Kilcast & Subramaniam, 2000). Beforehand, the type and number of subjects are determined based on availability of panelists, time allocated for training, and sensorial complexity of food product subject to analysis (Kilcast & Subramaniam, 2000). In addition, the type of test performed can dictate the type of statistical analysis required. For example, if only two samples are tested, a simple t-test can suffice but if there are more than two samples are tested and multiple attributes are measured by the panelists, more sophisticated statistical analysis is needed. These can include multivariate analysis and response surface methodology.

Panelists can be recruited from within a company, university department, or local population. Also, dedicated part time panelists can be recruited from a pool of individuals in a workplace environment. Once panelists are selected, they must be screened for familiarity of product as well as identification and detection of key sensory attributes. Screening tests are utilized to verify that sensory impairment is absent, establish sensitivity to appropriate stimuli, and evaluate ability to verbalize and communicate responses (Kilcast & Subramaniam, 2000). These screening tests depend mainly on defined objectives of sensory testing and typically consist of the following: (Kilcast & Subramaniam, 2000)

- Ability to detect and describe the four basic tastes of sweet, sour, salt and bitter. However, additional detection may be extended to include metallic, and astringent.
- Ability to detect and recognize common odorants coupled with odor characteristics of product range of interest.
- Ability to rank increasing intensities of a specific stimulus correctly. For instance, increasing sweetness or increasing firmness.
- Ability to characterize textural terms of relevant food types.
- Tests conducted using Ishihara charts to evaluate the absence of color vision deficiencies.

Acceptable performance across the entire range of tests merits selection of suitable panelists rather than excellent performance in some areas of test and poor performance in other areas. Nevertheless, if a sensory panel is to be utilized for a specific purpose (ex. detection of a single texture attribute) then screening tests relevant to that specific purpose can be weighted appropriately (Kilcast & Subramaniam, 2000). After panelists are screened, training is conducted to immerse panelists with specific product and product attributes of interest. Specifically, targeted training is conducted using the product(s) of interest with major emphasis directed towards specific tests to be used in practice. During practice or training, close monitoring of panel performance is essential to a successful completion of training program. Any noticed drift in panelist performance must be corrected by retraining procedures (Kilcast & Subramaniam, 2000).

After panelists have been selected, screened and trained, they are equipped for conducting sensory analyses according to specific trained method. There are a variety of methods utilized in conducting sensory evaluation. Many available test methodologies fall into two main classes. They are analytical tests and hedonic/affective tests. Analytical tests are used to measure sensory characteristics of products by providing answers to questions such as is there a difference, what is the nature of the difference(s), and how big is/are the differences(s). (Kilcast & Subramaniam, 2000) Hedonic/affective tests are used to measure consumer response to sensory characteristics of relevant product(s) by providing answers to the questions; which product is preferred and how much is it liked. Analytical tests are further classified into difference and quantitative tests. Difference tests include paired comparison, duo-trio, triangle and R-index, while quantitative tests include simple descriptive, profiling, and time-intensity.

Hedonic tests include preference acceptability and relative to ideal (Kilcast & Subramaniam, 2000).

For the current research study involving shelf life of celery in bio-based materials, the sensory test methodology utilized was a 15 cm unstructured scale. An unstructured scale requires panelists to be familiar with the range of attributes under study. Samples are presented and rated according to attributes listed on sensory ballot sheet. The 15 cm unstructured scale is a line anchored at both ends by ½ in. marks representing the terms that define and limit a specific attribute (Robertson, 1993). For instance, the texture attribute of a celery product can be limited by the anchor words, rubbery and crisp. In ranking that specific attribute for celery, the panelist simply marks across the line at the point which represents what is perceived. That specific point as a measurement of panelist perception is a data point which is measured as the distance from the left anchor mark to the panelist's mark. Other data points are collected in an identical manner for additional attributes and analyzed statistically. Analyses that can be conducted include Analysis of Variance, t-tests, and multiple comparison tests to identify significant differences in attributes within samples over time.

CELERY TEXTURAL ATTRIBUTES AND MEASUREMENT

Crisp and firm tissues are desirable textural qualities in vegetable crops such as celery. While some textural components can be evaluated visually via indications of wilting or shriveling, a plethora of other textural components for a wide array of produce require mechanical measurements to measure textural properties. Textural testing equipment, known by their manufacturer's names, such as Instron and Texture Technologies, are commonly used for evaluating various textural components of plant or vegetable tissues which change over time during storage or shelf life study. For instance, the softening of a product over time during a storage study can be detected mechanically via a texture measurement system equipped with artificial jaws attached to force gauges that can simulate bite action and ultimately evaluate textural qualities such as crispness, firmness, or softness (Kilcast & Subramaniam, 2000).

Textural attributes of celery must initially be identified prior to texture analysis in order to select appropriate probes that can accurately assess specific textural attributes relevant to a product. For instance, a good quality celery product exhibits turgor pressure, characterized by the rigidity of cell caused by outward pressure of water (Vina & Chaves, 2003). Consequently, the key textural attributes of celery to assess during texture analysis must encompass attributes that emphasize textural strength of the celery product. Moreover, a marker of a poor textural quality celery product indicates a product that is brittle and tough within interstitial fibers (Lamikanra, Imam, & Ukuku, 2005). Therefore, a major textural attribute such as firmness must be assessed as an effective indicator of textural quality.

Firmness, a good indicator of textural quality can be measured instrumentally with a texture analyzer such as a TA-XT2 plus texture analyzer fitted with a specific probe that makes contact with product during analysis in order to provide firmness data over time (Prakash, Inthajak, Huibregtse, Caporaso, & Foley, 2000). Additional texture analysis with a stainless steel probe can also measure the maximum shear force (MSF) applied before breaking (Rizzo & Muratore, 2009). Prior research involving texture of celery have utilized an Instron electromechanical testing system fitted with a Warner-Bratzler knife to measure the maximum shear force (MSF) in Newtons (N) applied before breaking. A 500 N transductor employed with a knife at a displacement rate of 10 mm per minute was used to measure the MSF on 20 different stalks that were cut transversally between ribs of the collenchyma (outside layer) and parallel to the fibers (Vina & Chaves, 2003). The complex and diverse structure of celery pertaining to ribs, curvature, variation in stalks, varying sizes, etc presents difficulties in arriving at a uniform textural analysis. Nevertheless, researchers cited in this study have utilized an approach of employing probes that make parallel contact between the ribs and penetrate downwards until completely puncturing samples.

Further textural analysis pertaining to shelf life studies have been conducted by researchers studying the effect of various factors on celery shelf life and quality. An analysis on texture of celery was performed by Prakash et.al in (2000) to measure firmness of gamma irradiated celery samples in a research study aimed at extending shelf life of precut celery. The firmness of treated samples were measured at room temperature by a puncture test using a Stable Micro Systems Texture Analyzer (model TA-XT2, Texture Technology Corp. Scarsdale, NY., U.S.A.) equipped with a 2 mm diameter stainless steel cylinder probe (TA P/2). The probe was set at 30 mm from the bottom of the plate and moved downward at a speed of 5 mm/s, stopping upon sensing samples, followed by further descending at 3 mm/s until completely puncturing samples (Prakash, Inthajak, Huibregtse, Caporaso, & Foley, 2000).

Firmness dominates as the key textural attribute to measure while utilizing probes such as the Warner-Bratzler. Besides the Warner-Bratzler knife probe, other probes have also been utilized such as a stainless steel cylinder probe, and a knife blade. Such probes are set at a specific distance from the bottom of the texture analyzer base and triggered downward at a constant speed to make contact with sample followed by further decrease at constant rate until completely puncturing samples. Afterwards, firmness is reported as the maximum force in grams and registered on a spreadsheet provided by instrument software (Seow, NG, & Bourne, 1992).

Other textural attributes measured in this study include toughness and stiffness. Both attributes are related to the structure and textural properties of fresh produce that is maintained by adequate cell turgor. Cell turgor is defined as the rigidity of cell wall caused by outward pressure of water content of each cell on membrane Kilcast, D., & Subramaniam, P. (2000). Generally, turgor pressure maintains rigidity of cells and nearby tissues while the stiffness and toughness assessments serve as complimentary mechanical measurements of turgidity. In deriving firmness, the incisor blade (probe) mounted onto the texture analyzer measures the peak force required for the blade to puncture a sample, while stiffness is derived by measurement of how fast the force (per distance) increases as probe penetrates sample. In other words, as a sample is masticated (chewed) by the teeth, the force required to masticate increases along with stiffness. Finally, the textural attribute of toughness is derived by measuring the energy required to masticate a sample.

COLOR OF VEGETABLE PRODUCTS

Color is a surface and aesthetic indicator of vegetable quality (Bartz & Brecht, 2003). A major feature in vegetable appearance, color can also function as a measure of physiological maturity, ripeness, and also an indicator of physiological, mechanical, or pathological injury (Bartz & Brecht, 2003). Alternatively, color can be a misleading gauge of vegetable quality if appearance is utilized as the sole marker of vegetable quality at the expense of other quality attributes such as flavor and texture. Nevertheless, color is a primary means of evaluating vegetable quality within the postharvest handling chain and in a consumer's decision to purchase a vegetable product. Vegetable products that look unappealing are highly unlikely to be purchased and consumed. Thus, method(s) of accurately measuring vegetable color in addition to understanding the effect of color on consumer acceptability is important in maintaining and enhancing vegetable quality and marketability (Bartz & Brecht, 2003).

Vegetable color can be quantified by colorimetry. Colorimetry is an instrumental technique that describes color mathematically in terms of human perception (Hunter and Harold; 1987 Hutchings, 1994; Shewfelt, 1993). Figure 1.5 identifies the most extensively used color scale based on the CIE color solid (L*a*b*). The color solid is based on the color opponent theory of expressing color of objects in terms of red-green character and blue-yellow character (Bartz & Brecht, 2003). Therefore, color that is more red then green is represented by "+a*," while more green than red is represented by "-a*." Additionally, color that is more yellow than blue is represented by "+b*," and more blue than yellow is represented by "-b* " (Bartz & Brecht, 2003). The L* color value of the color scale represents lightness in which 0 corresponds to black and 100 corresponds to white (Hunter, 1987).

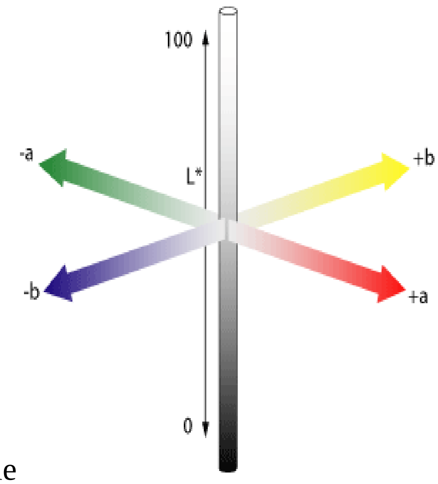

Figure 1.5: L* a* b* color scale
(Cooksey, 2007)

Prior research involving shelf life of celery has utilized the above color scale and values as a metric for measuring color quality over time. Along with the above color scale, a Hunter Lab Colorimeter (Model D25 PC2 Colorimeter, Reston, VA., U.S.A.) calibrated with black and white standard color tiles have been used to obtain color measurements. Results are recorded using CIE L, a, b color values (Kovacs, Horvath, & Bnecze-Bocs, 1977). Additionally, total color difference (ΔE) between any two time intervals were determined by measurement of individual points and calculated by equation 4 below:

$$\Delta E = [(L- L_0)^2 + (a-a_0)^2 + (b-b_0)^2]^{1/2}$$

(Prakash, Inthajak, Huibregtse, Caporaso, & Foley, 2000)

In the above formula, L_0, a_0 and b_0 represent color readings on the first day.

High quality celery consists of petioles (stalks) that are thick, compact, straight, and light green in color (Suslow & Cantwell, 1998). Nevertheless, there are variations in celery color within the outer middle and inner stalks. The middle and inner stalks tend to exhibit lighter shades of greenness than the outer stalks. Due to such disparity in shades of greenness from outer to inner stalks, measurements can be taken from outer, middle, and inner stalks in obtaining color values that are a fair representation of an individual celery quality. Rizzo et al obtained surface color of celery with a hand held colorimeter (NR-3000, Nippon Denshoku Ind. Co., Ltd., Japan) calibrated with a standard white tile with the following parameters: X=83.47, Y=84.43, Z=95.16. Using the ΔE formula mentioned above, L* a* b* data were collected on the internal, medium, and external stalks (Rizzo & Muratore, 2009).

CHAPTER 3

MATERIALS AND METHODS

Celery arrival and preparation

After harvest at marketable ripeness, 14-16 inches in stalk length (Luo, Suslow, & Cantwell, 2002) in Uma, Arizona, celery was brought to a packing house within close proximity to harvest fields. In the packing house, celery was pre-cooled via hydro-cooling to approximately 0°C followed by packaging into polyethylene sleeves (Figure 1.6) and boxed for transportation in refrigerated (1°C) trucks. The refrigerated trucks transported (4-5 days) sleeved celery in corrugated cases to a local warehouse in Greenville, South Carolina. At the warehouse, 14 cases of celery, 210 individual bunches (as ordered for research) were offloaded to smaller refrigerated trucks and transported (40 minutes) to Clemson University Packaging Science Department where the shelf life research was undertaken.

The shelf life research included a sensory analysis evaluation of refrigerated celery stalks (Figure 1.6). In preparation for sensory evaluation, leaves and 4-cm long segments of the basal plate of celery were removed with a knife in order to obtain unbranched petioles (Figure 1.7).

Figure 1.6: Sleeved Uncut Celery Stalk (Tanimura & Antle, 2007)

Figure 1.7: Cut Celery Stalk (Tanimura & Antle, 2007)

Petioles were washed with tap water while brushing thoroughly to remove any dirt or debris. Afterwards, they were allowed to dry under room conditions followed by sorting for uniformity and freedom of defects. Visual quality attributes were examined for appearance and microbial spoilage. Celery sticks with extreme defects were immediately discarded. A defected celery stick was one containing enough defects to prevent attaining of at least four 4 cm cut samples. After being completely dried, samples were prepared for sensory evaluation by cutting with a sharp clean knife into 4-cm long strips.

PACKAGING MATERIALS

Celery stalks were packaged into four different materials. The control material was Low Density Polyethylene (LDPE) which contained perforations and was provided by the celery supplier, Tanimura and Antle. The remaining three variable materials were un-perforated and were supplied by Innovia Films Ltd. The variable materials arrived in rolls and were cut into individual sheets that matched the dimensions of the control material, 2 × (42 × 17 cm). After being cut, they were folded over length wise to match the exact dimensions (42 × 17 cm) of the control material followed by low temperature sealing (~ 5 seconds) on sides and bottom with an Onor Pack hand operated heat sealer (Model FS-200).

All four materials, along with their respective thicknesses, permeabilities, and dimensions are listed in table 1.1 below. Additional information contained in specification sheets can be found in the appendix.

Table 1.1: Material OPR, WVPR, Thickness & Dimensions

OPR	OPR: cc· mil/ (m^2 · day) 754.17 mmHg @ 23.0°C			
	LDPE	Ecoflex	Mater-Bi	PLA
	9500	2793	2673	494
WVPR	WVPR: g · mil/ (m^2 · day) 754.17 mmHg @ 23.0°C			
	LDPE	Ecoflex	Mater-Bi	PLA
	24	995	647	211
Thickness (µm)	LDPE	Ecoflex	Mater-Bi	PLA
	1.24	1.07	1.26	1.06
Dimensions (cm)	LDPE	Ecoflex	Mater-Bi	PLA
	42×17	42×17	42×17	42×17

Material Thickness Measurement

Material thickness was measured with a micrometer (L.S. Starrett Co., of Athol MA, USA) to the nearest 0.001 mm at 3 random positions on films.

Material Analysis

<u>Permeation</u>

Oxygen permeation and water vapor permeation rates of films were determined using ASTM method D-3985 and F-1249 for oxygen and water respectively. Testing conditions were at 23°C and 100%RH. All samples were masked with aluminum in order to avoid failures during testing. Sample sizes were cut at $100m^2$ and 50 m^2 for oxygen and water vapor respectively.

Oxygen Permeability Rate (OPR)

Oxygen permeability was determined according to ASTM Standard Method D 3985 on each material at weeks 0, 6 and 12. Oxygen permeability was measured by using a Mocon Ox-Tran 1000 Permeability Tester (Modern Control Inc, Minneapolis, MN). For each material, duplicates samples were masked and mounted onto the Ox-Tran followed by conditioning for 12-18 hours with nitrogen gas. Testing was performed at 23°C and 100%RH. To calculate OPR of tested specimen, derived OPR was multiplied by thickness of tested film.

Water Vapor Permeability Rate (WVPR)

WVP of duplicate film samples (also masked) were determined at 23°C and 100% RH. Films were tested using The Permatran-3/31 (Modern Controls, Inc. Mocon, Minneapolis, MN) according to ASTM Standard Method F-1249 (ASTM, 1990). Test cells were mounted by masked samples of each material. One cell contains a fixed amount of moisture while the other is connected to a humidity sensor that is able to determine moisture content in dry purge air (Robertson, 1993). Meanwhile, the relative humidity of air surrounding the humidity sensor is elevated via the movement of water vapor through the film, enabling the recording of time for a given rise in relative humidity (Robertson, 1993).

Tensile Tests

Tensile tests were conducted according to ASTM Standard Method D-882 for Tensile Properties of Thin Plastic Sheeting (ASTM, 1991) on an Instron Universal Testing Machine (Model 4201, Instron Corp., Canton, MA, USA). Three samples of each film type were cut to 4 inches in length before testing. Each strip of film was placed between pneumatic jaws of the Instron that was preset to 51 mm (ASTM, 1991). Film strips were stretched at a rate of 30 mm per minute until sample failure. Measurements of load (N) and deformation (mm) were used to calculate tensile strength and elongation to break. Tensile strength was calculated by dividing maximum load placed on sample by the cross-sectional area while elongation to break was calculated by dividing deformation of sample at maximum load by the original gauge length (ASTM, 1991). Relevant equations can be viewed in the literature review section.

SENSORY EVALUATION OF CELERY

Sensory evaluation was conducted with 15 panelists familiar with the product. Panelists participated in two 30 minute training sessions to identify and evaluate key celery sensory characteristics of color, off aroma, firmness, and celery flavor (Prakash, Inthajak, Huibregtse, Caporaso, & Foley, 2000). During training, panelists indentified celery attributes relating to aroma, appearance, flavor and texture. Their identification of those attributes were tailored to depict anchor words on opposite ends of 15 cm unstructured scale used for sensory scoring. For instance, after panelists identified firmness as a key textural attribute, anchor words tailored to reflect firmness were depicted on sensory scoring sheet as 0 = rubbery 15= crisp.

A 15 cm unstructured scale was used to rank the following attributes: aroma, appearance, flavor, liking of flavor, texture, overall liking and sample rank. The lower end of scale corresponded to undesirable quality descriptors while higher end corresponded to desirable quality descriptors. For instance, the appearance attribute contains anchor descriptors of old and fresh, equivalent to anchors 0 and 15 respectively. Celery samples (4 cm length) were given to panelists in cups coded with 3 digit random numbers. Samples were evaluated biweekly (week 0, 2, 4, 6, and 8) for a total of 8 weeks during storage. After week 8, microbial levels (aerobic bacteria) surpassed 7 Log CFU/g, rendering samples unsafe for consumption.

COLOR ANALYSIS OF CELERY

Color analysis was determined using a Konica Minolta CR 300 colorimeter with an 8 mm diameter measuring area and calibrated (L* 95.52, a* 0.06, b* 1.81) with a standard white plate. Measurements were conducted by applying the colorimeter head on the outer surface of celery sticks to obtain the L*, a* and b* values of the CIE scale. Measurements were taken on three different spots on the outer surface of celery stalks. For each sample, three stalks, as depicted in Figure 2.8, (inner, middle and outer) were used to obtain color measurements.

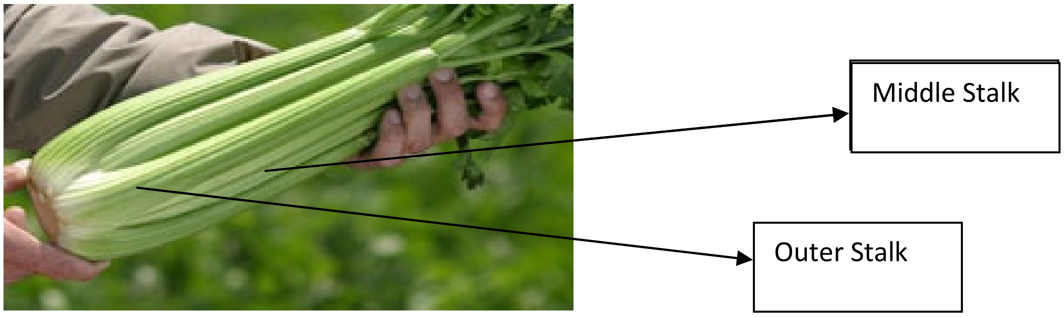

Figure 2.8: Celery Stalk

Measurements were expressed as L*, a* and b* parameters. The parameter ΔE recognizes total color change by utilizing equation 4 below:

$\Delta E=[(L_f - L_i)^2+(a_f - a_i)^2+(b_f - b_i)^2]^{0.5}$ (Prakash, Inthajak, Huibregtse, Caporaso, & Foley, 2000)

L_i, a_i, & b_i represent readings at week 0, while L_f, a_f, & b_f represent readings at each other subsequent week.

TEXTURE ANALYSIS OF CELERY

Texture analysis was performed by measuring three different textural attributes using a TA-XT2 plus texture analyzer fitted with a TA-45 Incisor Knife Blade, 1.5 mm wide x 11 mm long. Measurements were made on three celery stalks per material. For each celery sample, three stalks (outer, middle, and inner) were subjected to five Incisor Knife Blade penetrations to produce data on firmness, stiffness, and toughness. Sticks were penetrated horizontally along the length of celery. Distance from celery contact surface to tip of blade was at 1 cm with a penetration depth of 0.05 mm. The measurements provided data on Force (Kg), Gradient

(Kg/sec), and Area (Kg·sec), which corresponds to textural attributes of firmness, stiffness and toughness respectively.

WEIGHT LOSS OF CELERY

Weight measurements were conducted with a standard balance (Scout Pro. Model SP4001). Weight loss was determined by weighing the same samples designated specifically for the weight analysis category. The same set of samples per material were weighed during each week using a simple formula, % weight loss = {*(I-F)/I} *100*, and results were expressed as a percentage. I and F denotes initial and final respectively. Initial weight always denotes weight at week 0.

MICROBIAL QUALITY OF CELERY

Total aerobic plate counts were (triplicate plates) used to analyze microbial quality of celery samples 48 hrs before sensory evaluation. An 11 gram sample from celery packaged in each material was homogenized for 2 minutes in 99mL of sterile peptone water in a sterile stomacher bag with a stomacher (Seward Stomacher 400 Circulator). Plate count agar was used for bacteria enumeration after 48 hrs in incubation at 38°C. Microbial counts were expressed as $\log_{10}$ CFU g^{-1}. Samples were analyzed at week 0, 2, 4, 6, and 8. The maximum aerobic bacteria limit is10^7 CFU g^{-1} (Gomez & Artes, 2005). After week 8, microbial levels (aerobic bacteria) surpassed 7 Log CFU g^{-1} and microbial analysis as well as sensory evaluation was discontinued.

STORAGE CONDITIONS

Celery stalks packaged in the different materials were stored in refrigeration conditions (Miracool^{TM} Cooler Unit, Model 79BC690004-02) at 5°C & 95% RH and analyzed bi-weekly for a period of 3 months. Samples were analyzed at week 0, 2, 4, 6, 8, 10 and 12, except for sensory and microbial analysis which were discontinued after week 8. Storage experiments were represented as shown in Tables 1.2 and 1.3.

TABLE 1.2: EXPERIMENTAL DESIGN: Part A

Analysis	Ecoflex	LDPE	Mater-Bi	PLA
Appearance (Color & Pics)	3	3	3	3
Sensory, Micro, & Material Analysis	3	3	3	3
Texture	3	3	3	3
Weight (reused)	3	3	3	3
TOTAL	12	12	12	12

3= # Celery Bunch & # Replicates

12 Bunches to satisfy each material & all four analyses

TABLE 1.3: EXPERIMENTAL DESIGN: Part B

# Sleeves (bags) per Material	Week
48	0
36	2
36	4
36	6
36	8
36	10
36	12
240: Total # Sleeves	

*After week 0, the 12 celery stalks for weight analysis were reused for remainder of study. Therefore, only 36 (48-12) were used for other analyses.

STATISTICAL ANALYSIS

Statistical analysis was conducted based on time (week) and material. Data were treated by Analysis of Variance (ANOVA). Comparison of means pertaining to all analyses conducted within weeks were subjected to Fisher's Least Significant Difference (LSD) test at a significance level $p = 0.05$.

CHAPTER 4

RESULTS & DISCUSSION

The objective of this research was to determine if biopolymer films could be used to package fresh celery and if they were comparable to the currently used packaging material (LDPE). Chapter 4 provides results of the objective and subjective testing of celery as well as properties of the packaging materials during the 12 week storage period.

Table 4.1: Weight loss (%) of celery during refrigerated storage

Material	week 0	week 2	week 4	week 6	week 8	week 10	week 12	Overall
Control	0.00 a	4.25 a	8.72 a	13.7 b	18.7 b	23.3 a	26.5 a	13.6 b
Ecoflex	0.00 a	4.0 a	6.5 a	9.2 a	15.3 a	20.1 a	24.0 a	11.3 a,b
Mater-Bi	0.00 a	3.0 a	5.7 a	9.0 a	12.0 a	16.0 b	19.6 b	9.3 a
PLA	0.00 a	2.81 a	5.7 a	9.0 a	13.6 a	20.7 a	23.6 a	10.8 a

*Within columns, materials with same letter are not significantly different

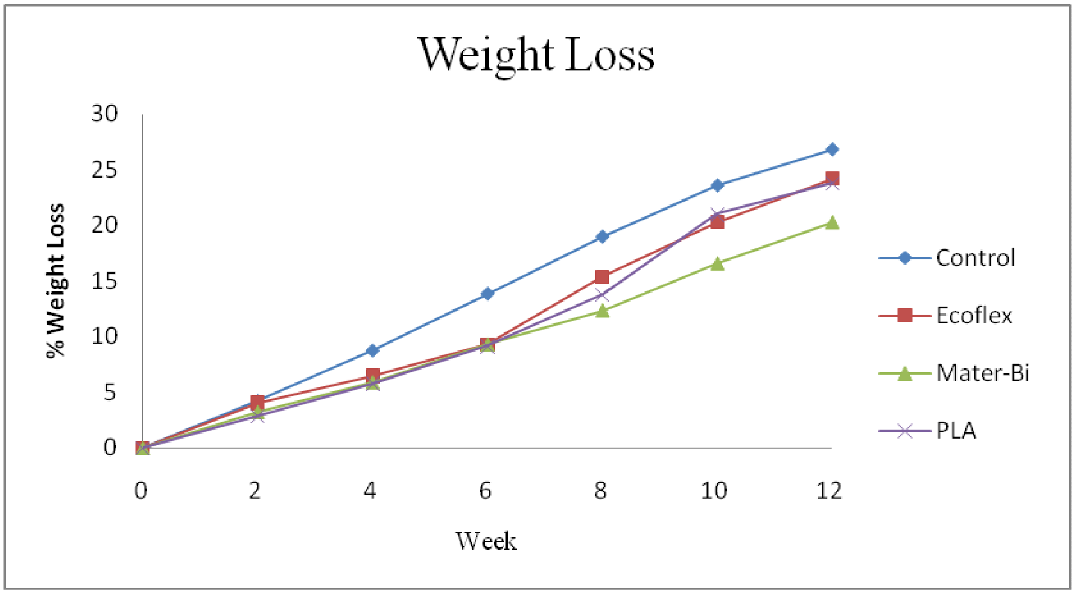

Fig 4.1 % Weight loss (%) of celery during refrigerated storage

Celery contained within all materials experienced a decrease in weight over time. The Mater-Bi material experienced the least decrease in weight while the control material experienced the greatest weight loss. Overall, both materials were significantly different ($p<0.05$) from each other, while Mater-Bi, PLA and Ecoflex were not ($p>0.05$). Weight loss was statistically different between materials beginning in week 6. Celery in the control material lost significantly more weight than celery contained in other materials between weeks 6-8. Over entire 12 week period, Tukey's Test ($p>0.05$) identified Mater-Bi material as significantly different from the control in experiencing less percent weight loss.

Packaged celery in the control film may have lost more weight than celery packaged in other films because it contained perforations while the other films did not. In comparing performance with regard to weight loss amongst the biopolymers, celery packaged in the Mater-Bi material experienced the least percent weight loss. Mater-Bi was the thickest of all the biopolymer materials and also contained some hydrophobic polymers blended with corn starch during manufacturing. These characteristics may have improved moisture retention and mechanical properties of Mater-Bi, thereby rendering it as the best performer of bio-based materials in this regard.

Rizzo and colleagues utilized a polyolefin material with an antifogging additive (AF) and micro perforated polypropylene (MP) in studying the effects of packaging on the shelf life of fresh celery. The AF was a non-perforated co-extruded polyethylene and polypropylene film treated with an antifogging additive, while the MP was a co-extruded polypropylene with a piercing density of 7 holes/cm^2. Celery was stored at 4°C, 90% RH for 35 days. A set of unpackaged celery was stored as a control. As expected, weight loss increased during storage for both plastic films. In the MP film, weight loss ranged from 10.7% at day 6 to 33.6% at day 31, which was similar to the unpackaged control. Weight loss of celery in the AF package was lower than 3 % over the 31 day period while the control (unpackaged) had 27% weight loss over 20 day period. The antifogging layer was critical in preventing accumulation of condensate on film surface that could enhance moisture loss and reduce shelf life. By day 17, unpackaged celery significantly lost more weight than MP film due to an absence of a film to entrap moisture or prevent excess moisture loss during transpiration.

At week 2, celery packaged in the AF material was significantly lowest in weight loss at 0.83% compared to celery packaged in the MP film (19.66 %) and unpackaged celery as control (24.24 %). However, at week 2 in current study, celery packaged in PLA material was lowest in weight loss at 2.8% but was not significantly different than weight loss of celery packaged in other materials. In a two week weight loss comparison between both studies, the AF material utilized by Rizzo and colleagues demonstrated better moisture retention than materials utilized in current study. However, materials utilized in current study performed better at moisture retention during a two week period comparison to the MP material utilized by Rizzo and colleagues.

Color

Table 4.2 ΔE Analysis: Outer Stalks

Material	week 0-2	week 0-4	week 0-6	week 0-8	week 0-10	week 0-12
Control	2.75	8.75	12.08	17.84	17.61	19.36
Ecoflex	5.36	12.31	18.84	16.54	20.75	23.88
Mater-Bi	2.49	5.56	13.39	13.46	17.45	21.95
PLA	2.10	3.24	15.41	15.80	17.23	18.49

*Within columns, materials with same letter are not significantly different

*ΔE is calculated through a formula and takes into account week 0 and each other subsequent week. Consequently, one data point is obtained and statistical analysis cannot be achieved.

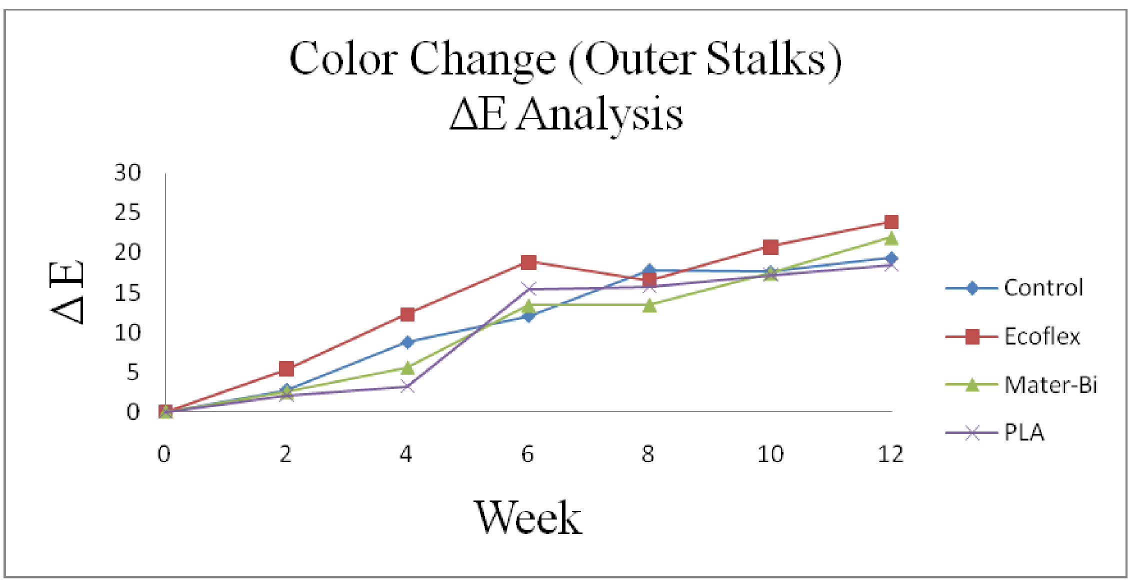

Figure 4.2: ΔE Outer Stalks

Celery packaged within all materials experienced color change throughout the 12 weeks of storage. The celery packaged in Ecoflex material appears to experience a greater degree in color change from weeks 0-6 but eventually levels off along with other materials thereafter. However, since statistical analysis could not be performed, it cannot be determined if these differences are significant.

Over time, the Ecoflex films under storage began to disintegrate. Consequently, the Ecoflex material was expected to perform poorly in comparison to other materials. However, no obvious impact on the color attribute was noticed.

ΔE Analysis: Middle Stalks

Table 4.3 ΔE Analysis: Middle Stalks

Material	week 0-2	week 0-4	week 0-6	week 0-8	week 0-10	week 0-12
Control	8.92	11.40	13.58	14.07	16.26	17.93
Ecoflex	2.33	5.59	9.02	7.95	13.12	16.11
Mater-Bi	5.12	3.29	7.10	10.33	12.17	13.54
PLA	4.37	1.44	9.90	8.80	14.05	14.09

*Within columns, materials with same letter are not significantly different

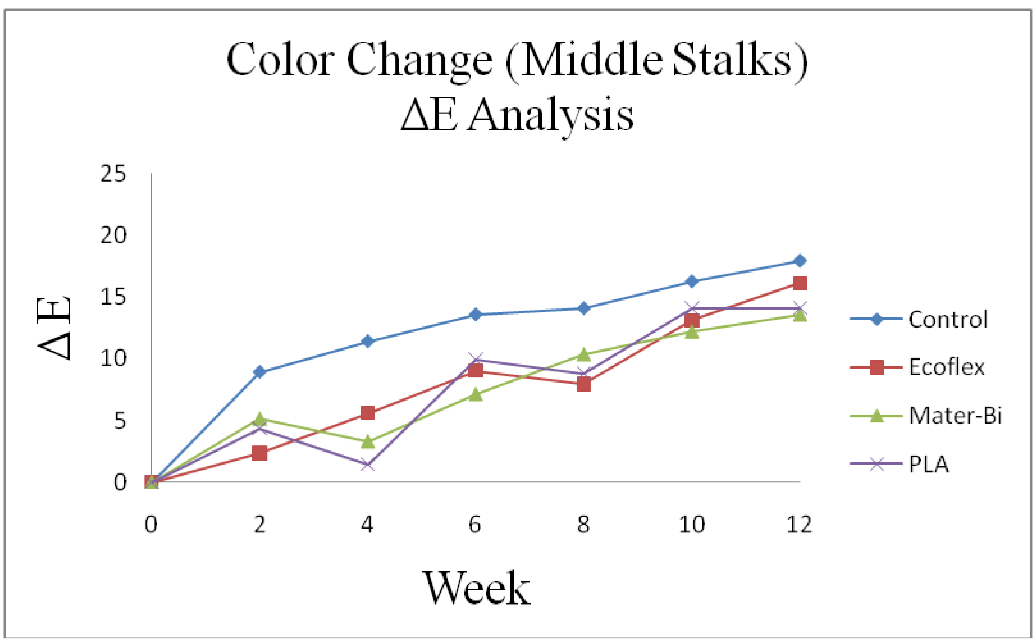

Figure 4.3: ΔE Middle Stalks

The Control material experienced a greater degree in color change over the 12 weeks of storage. The ΔE analysis for the outer stalks depicted Ecoflex showing a greater change in ΔE while control was showing a greater change in ΔE for the middle stalks. Such disparity in ΔE between outer and middle stalks is possibly due to inherent variability in celery product.

ΔE Analysis: Inner Stalks

Table 4.4 ΔE Analysis: Inner Stalks

Material	week 0-2	week 0-4	week 0-6	week 0-8	week 0-10	week 0-12
Control	4.46	4.86	12.13	11.36	10.96	13.14
Ecoflex	7.30	5.86	6.87	7.64	12.49	11.19
Mater-Bi	3.74	2.06	1.29	6.93	6.08	8.02
PLA	3.74	1.07	4.73	5.27	7.78	8.34

*Within columns, materials with same letter are not significantly different

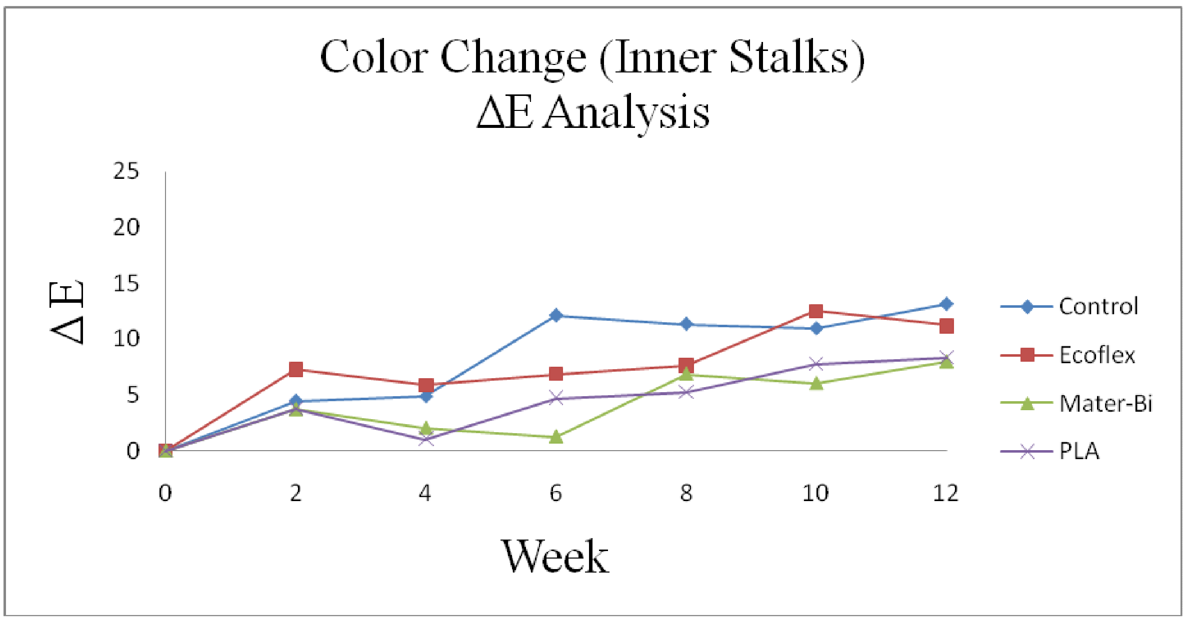

Figure 4.4: ΔE Inner Stalks

As expected, the inner stalks are lighter in color compared to outer stalks and do not reach higher ΔE values as the outer and middle stalks. The same variability and similarity in trend was also observed for the middle stalks.

L* Color Analysis: Outer Stalks

Table 4.5 L* Color Analysis: Outer Stalks

Material	week 0	week 2	week 4	week 6	week 8	week 10	week 12	Overall
Control	44.02 a	46.40 a	52.60 a	54.10 a	59.76 a	59.39 a	61.38 a	53.95 a
Ecoflex	41.40 a	46.60 a	53.27 a	58.33 a	54.08 a,b	60.03 a	63.77 a	53.93 a
Mater-Bi	41.27 a	43.65 a	46.50 b	54.0 a	52.06 b	57.27 a	61.85 a	50.95 b
PLA	43.10 a	45.0 a	45.66 b	56.85 a	57.06 a,b	58.41 a	59.74 a	52.26 a,b

*Within columns, materials with same letter are not significantly different

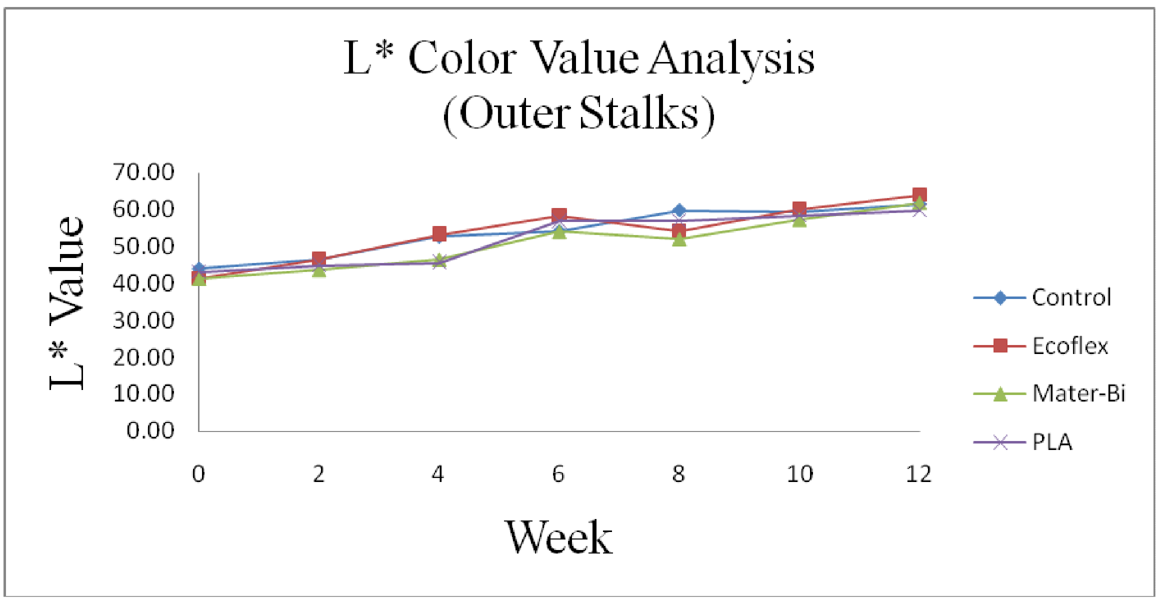

Figure 4.5: L color value. Outer Stalks

There were significant differences (p>0.05) between materials but only at weeks 4 and 8. Celery in Mater-Bi was identified as significantly lower in L* value from celery in Control and Ecoflex materials at weeks 4, 8 and for overall analysis. In general celery packaged in all materials progessively got lighter due to natural aging of product.

L* Color Analysis: Middle Stalks

Table 4.6 L* Color Analysis: Middle Stalks

Material	week 0	week 2	week 4	week 6	week 8	week 10	week 12	Overall
Control	47.0 a	55.77 a	58.34 a	60.50 a	60.95 a	63.03 a	64.10 a	58.53 ab
Ecoflex	53.18 a	55.48 a	57.96 a	61.35 a	60.65 a	65.25 a	66.51 a	60.05 a
Mater-Bi	50.70 a	55.75 a	53.70 b	57.60 a	60.91 a	62.50 a	62.83 a	57.71 b
PLA	50.84 a	55.0 a	51.60 b	60.50 a	59.48 a	64.32 a	63.07 a	57.83 b

*Within columns, materials with same letter are not significantly different

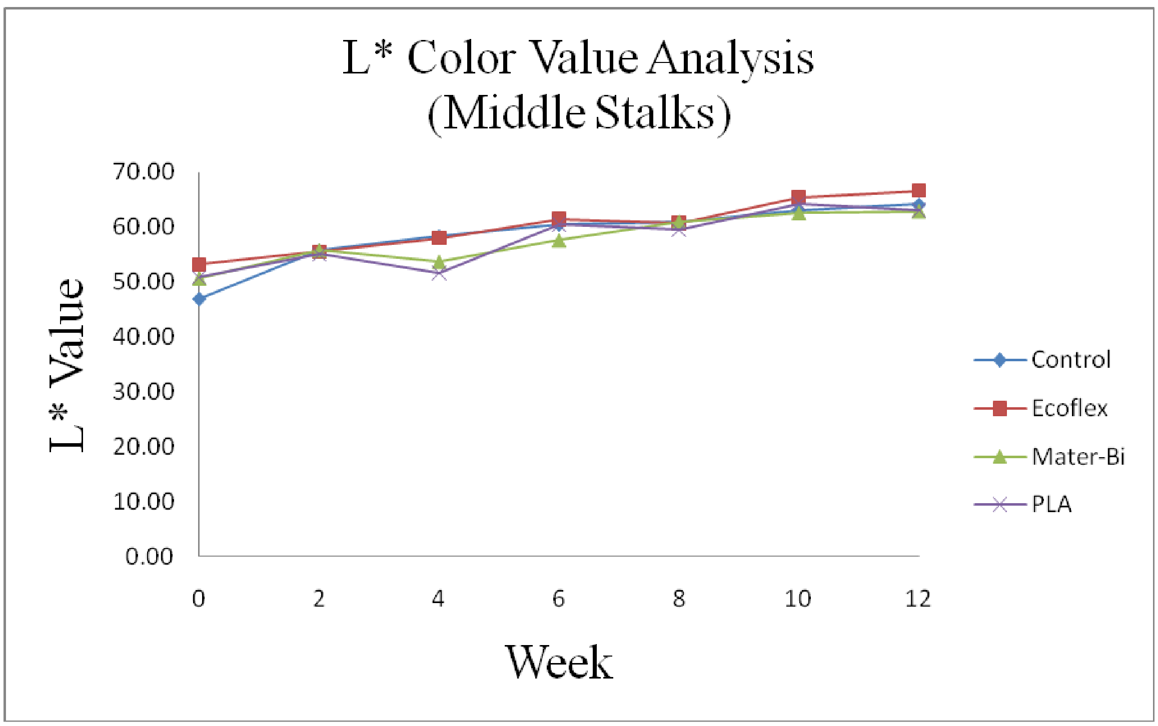

Figure 4.6: L color value. Middle Stalks

There were no significant differences between materials when comparing on a weekly basis except at week 4 when celery in PLA and Mater-Bi were statistically shown to be significantly lower in L* (lighter) than the celery in the Control and Ecoflex materials. Overall, celery in Ecoflex material was identified as significantly lighter than celery contained in Mater-Bi and PLA materials. Nevertheless, all materials followed a similar trend of steady increase in L* value from week 6-12. The trend for middle stalks follow a matching pattern to the trend for outer stalks.

Table 4.7: L* Color Analysis: Inner Stalks

Material	week 0	week 2	week 4	week 6	week 8	week 10	week 12	Overall
Control	52.09 a	56.46 a	56.58 a	63.90 a	63.25 a	62.97 b	65.04 a	60.04 b
Ecoflex	55.16 a	62.13 a	60.95 a	61.44 ab	62.76 a	66.40 a	65.20 a	62.00 a
Mater-Bi	58.71 a	58.33 a	57.86 a	58.94 b	64.15 a	63.51 ab	64.67 a	60.88 ab
PLA	58.0 a	59.68 a	57.19 a	62.22 ab	61.85 a	64.39 ab	65.51 a	61.26 ab

*Within columns, materials with the same letter are not significantly different

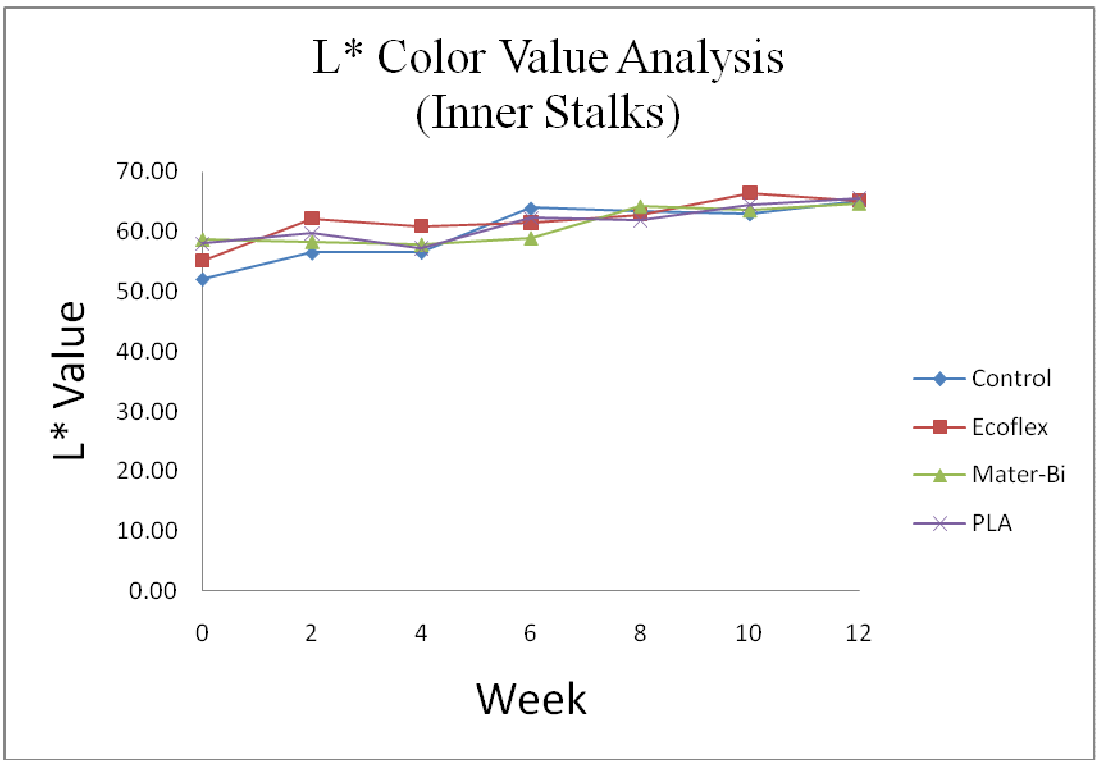

Figure 4.7: L* Color Value: Inner Stalks

Overall, celery in Ecoflex material was significantly lighter than celery in Control material. At week 6, celery in Control was significantly lighter than celery in Mater-Bi. Similarly, at week 10, celery in Ecoflex material was significantly lighter than celery in Control material. In general, the L* color value for outer, middle and inner stalks all follow a similar trend of increasing L* over time. As the weeks progress, the dominant pigment, chlorophyll, responsible for maintaining innate celery color began to fade due to natural deterioration, while carotenoids (pigments that coexist with chlorophyll), responsible for the lighter hue of yellow, began to dominate.

a* Color Analysis: Outer Stalks

Table 4.8: a* Color Analysis: Outer Stalks

Material	week 0	week 2	week 4	week 6	week 8	week 10	week 12
Control	-12.02 a	-10.66 a	-10.77 a	-5.69 a	-3.64 a	-3.75 a	-3.56 a
Ecoflex	-12.06 a	-11.33 a	-8.98 a	-3.89 a	-1.57 a	-3.40 a	-4.04 a
Mater-Bi	-11.40 a	-11.37 a	-9.55 a	-7.43 a	-3.40 a	-4.49 a	-3.90 a
PLA	-11.53 a	-11.48 a	-9.55 a	-4.77 a	-4.29 a	-3.71 a	-4.14 a

*Within columns, materials with same letter are not significantly different

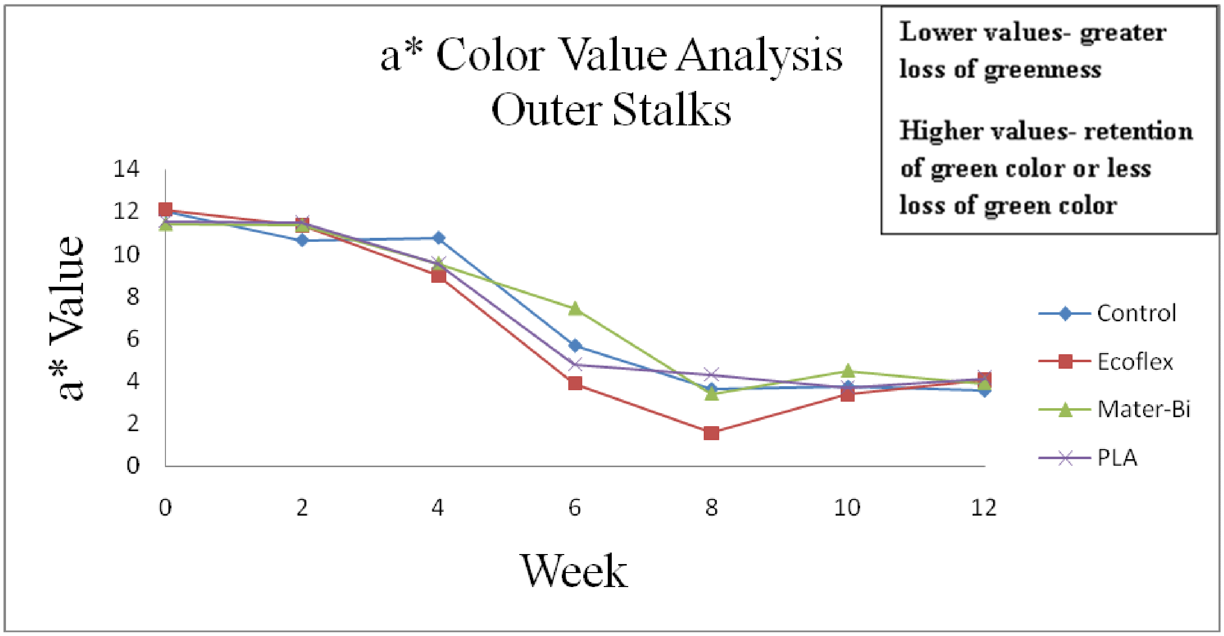

Figure 4.8: a* Color Analysis: Outer Stalks

Color measurement of a* values showed no significant differences between materials. The chlorophyll pigment responsible for green color naturally degrades during storage. Consequently, celery in all materials lost greenness over time. The a* color measurement resulted in lower values corresponding to greater loss of greenness and higher values corresponding to less loss of green color or a greater retention of green color.

a* Color analysis: Middle Stalks

Table 4.9: a* Color Analysis: Middle Stalks

Material	week 0	week 2	week 4	week 6	week 8	week 10	week 12	Overall
Control	-12.27 a	-12.75 a	-11.01 a	-10.81 a	-10.37 a	-9.55 a	-7.03 a	-10.54 a
Ecoflex	-12.78 a	-12.36 a	-10.77 a	-9.94 a	-10.43 a	-8.71 a	-5.34 a	-10.05 a
Mater-Bi	-12.71 a	-12.04 a	-13.03 b	-11.02 a	-11.14 a	-9.87 a	-7.26 a	-11.01 a
PLA	-12.84 a	-11.52 a	-11.70 ab	-10.80 a	-11.16 a	-9.22 a	-6.34 a	-10.51 a

*Within columns, materials with same letter are not significantly different

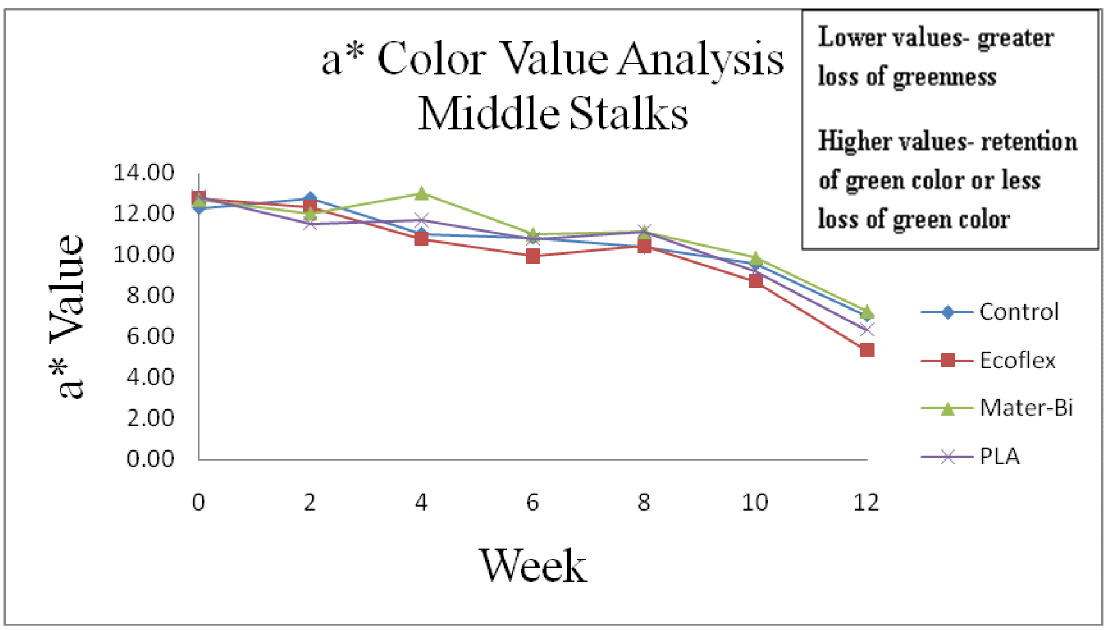

Figure 4.9: a* Color Analysis: Middle Stalks

At week 4, celery in both Control and Ecoflex materials experienced a significantly greater loss of greenness than celery contained in the Mater-Bi material. There was no other significant differences ($p>0.05$) between materials within each week, nor were there differences in materials overall. The a* color value for the outer stalk had declined sharply from week 0-8, while the same value for the middle stalk experienced a more gentle decline before sharply falling from week 10-12. The reason for such disparity in greenness between outer and middle stalks is because the outer stalks have a darker shade of greenness, while the middle has a lighter hue of green. Additionally, the outer stalks age faster than middle or inner stalks because a greater surface area containing chlorophyll, responsible for green color, is exposed to ambient conditions of oxygen and therefore degrade faster.

a* Color analysis: Inner Stalks

Table 5.0: a* Color Analysis: Inner Stalks

Material	week 0	week 2	week 4	week 6	week 8	week 10	week 12	Overall
Control	-11.93 a	-11.04 b	-10.40 a	-9.42 ab	-9.88 a	-10.67 b	-9.94 b	-10.47 b
Ecoflex	-11.22 a	-9.12 a	-10.41 a	-9.18 a	-10.61 a	-7.02 a	-7.01 a	-9.22 a
Mater-Bi	-12.30 a	-9.66 ab	-11.29 a	-11.53 b	-9.67 a	-9.75 b	-8.60 ab	-10.40 b
PLA	-11.94 a	-9.46 ab	-11.78 a	-10.29 ab	-9.72 a	-8.90 ab	-8.91 b	-10.14 b

*Within columns, materials with the same letter are not significantly different

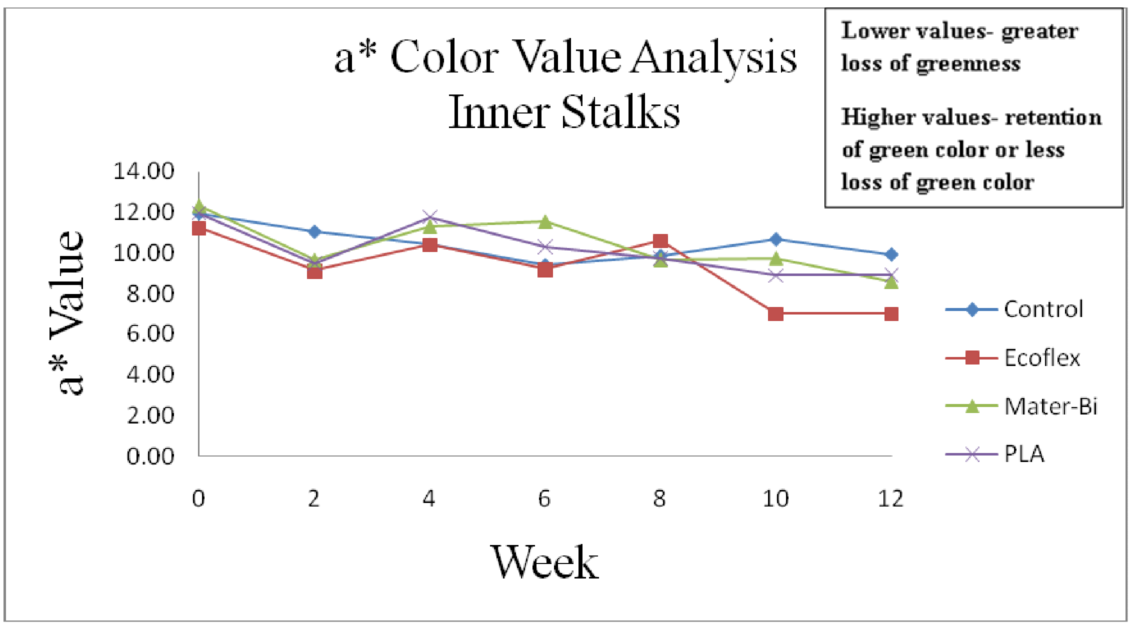

Figure 5.0: a* Color Analysis: Inner Stalks

Overall, celery in Ecoflex experienced greater loss of greenness than celery in other materials. There was no constant trend throughout the weeks but rather a random fluctuation (possibly due to inherent product variation) from week 0-12. However, within some weeks, there were significant differences in greenness values between materials. At week 2, celery in Ecoflex material experienced significantly greater loss of greenness than the Control but was not significantly different than celery packaged in other bio-based materials. Similarly, at weeks 10 and 12, celery in Ecoflex material also experienced significant loss of greenness compared to Control. Nevertheless, at weeks 0, 4, and 8, there were no significant differences in a* color value for celery packaged in each material. The inner stalks naturally vary with regard to color, compared to outer stalks, and therefore show greater variation in measurements over time.

b* Color analysis: Outer Stalks

Table 5.1: b* Color Analysis: Outer Stalks

Material	week 0	week 2	week 4	week 6	week 8	week 10	week 12	Overall
Control	17.17 a	17.27 a	18.32 a	15.10 b	17.25 ab	14.85 a	15.72 a	16.53 ab
Ecoflex	17.08 a	18.14 a	18.20 a	15.92 ab	15.50 ab	14.23 a	14.73 a	16.25 ab
Mater-Bi	16.64 a	17.37 a	16.23 a	17.86 a	17.57 a	15.78 a	15.17 a	16.66 a
PLA	16.71 a	17.63 a	16.56 a	15.06 b	15.21 b	15.59 a	13.46 a	15.74 b

*Within columns, materials with same letter are not significantly different

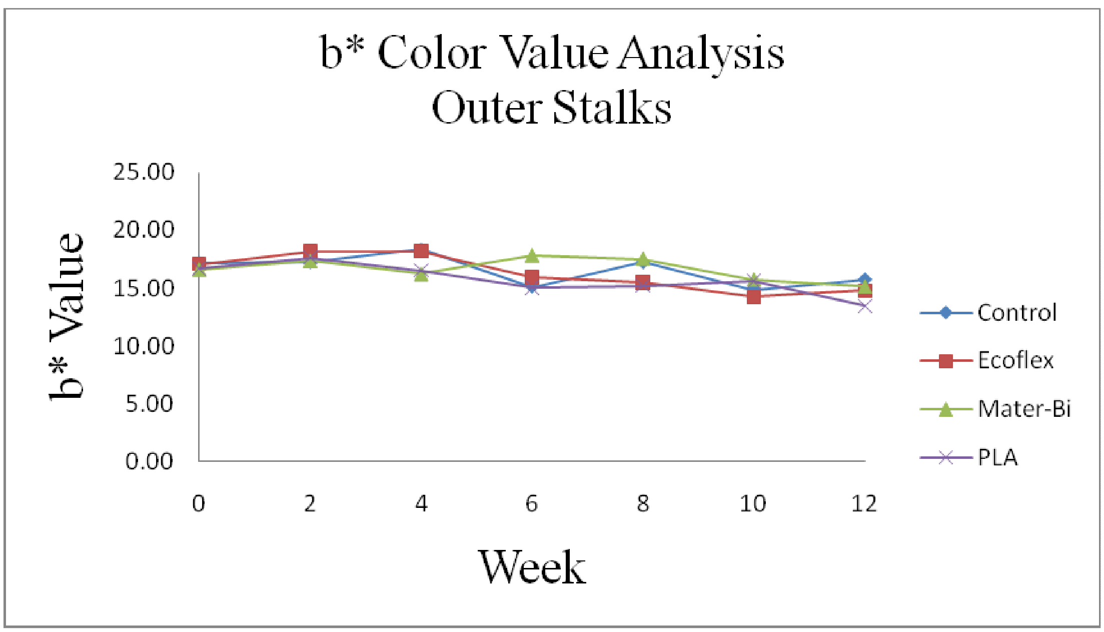

Figure 5.1: b* Color Analysis: Outer Stalks

The above b* color analysis is relevant with respect to yellowing of vegetables. At week 6, celery in Mater-Bi material experienced significantly greater yellowing than celery contained in PLA and Control materials. Similarly, the same development occurs at week 8 with celery in Mater-Bi experiencing significantly greater yellowing than celery in PLA. Plant pigments, such as carotenoids are primarily responsible for the yellow hue in vegetables after the fading of chlorophyll, responsible for the green color. A loss of green color (week 4) was found to be significantly greater for celery in the Mater-Bi material. Hence, greater loss of greenness prompts the dominance of a yellow hue attributable to yellow carotenoid pigments.

b* Color Analysis: Middle Stalks

Table 5.2: b* Color Analysis: Middle Stalks

Material	week 0	week 2	week 4	week 6	week 8	week 10	week 12	Overall
Control	18.52 a	20.16 a	18.33 b	19.04 a	18.65 a	18.20 a	17.23 a	18.6 a
Ecoflex	20.27 a	20.41 a	18.16 b	17.68 a	18.82 a	17.09 a	15.09 a	18.22 a
Mater-Bi	19.34 a	19.87 a	20.68 a	18.84 a	19.58 a	18.37 a	16.81 a	19.07 a
PLA	19.42 a	19.26 a	18.95 b	18.70 a	19.44 a	17.80 a	16.84 a	18.63 a

*Within columns, materials with same letter are not significantly different

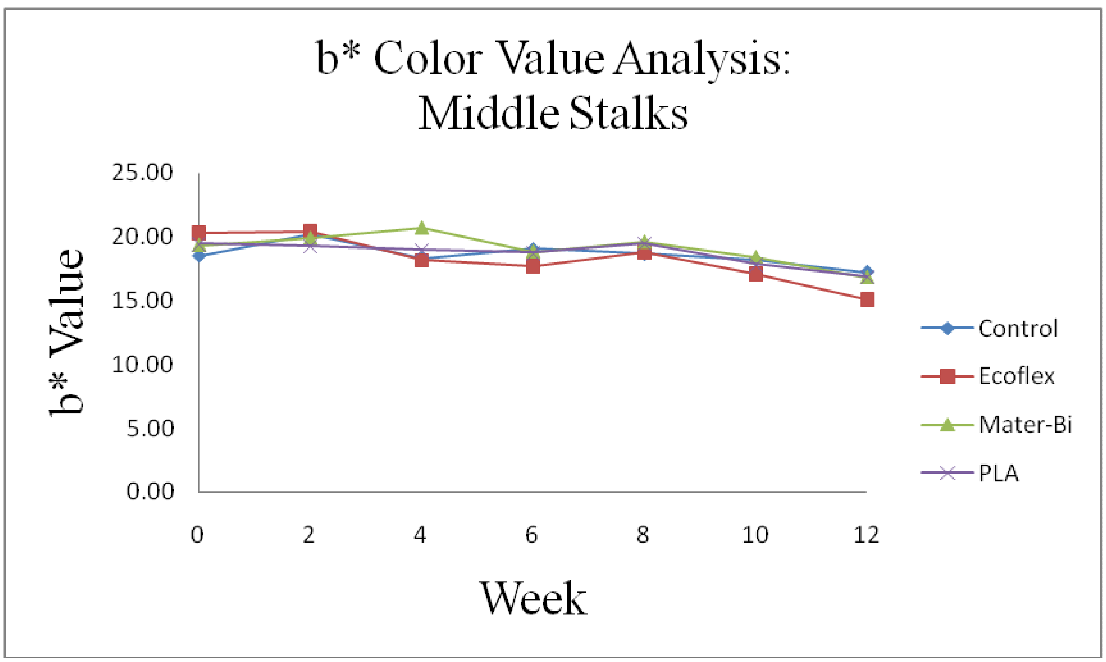

Figure 5.2: b* Color Analysis: Middle Stalks

There were no significant differences between materials within weeks nor were there differences in materials overall. Essentially, there was very little change in b* color value for the middle stalks throughout the storage period. The above trend is similar to the outer stalks for b* color analysis.

Table 5.3: b* Color Analysis: Inner Stalk

Material	Week 0	week 2	week 4	week 6	week 8	week 10	week 12	Overall
Control	19.24 a	19.34 a	18.15 a	18.01 ab	18.60 a	19.73 a	20.26 a	19.04 a
Ecoflex	19.06 a	18.58 a	18.66 a	17.14 b	19.50 a	15.58 b	16.46 c	17.85 b
Mater-Bi	20.86 a	18.24 a	19.28 a	19.85 a	17.47 a	18.14 a	16.96 bc	18.68 ab
PLA	20.55 a	18.30 a	19.86 a	19.21 ab	17.71 a	17.31 ab	18.55 ab	18.78 a

*Within columns, materials with the same letter are not significantly different

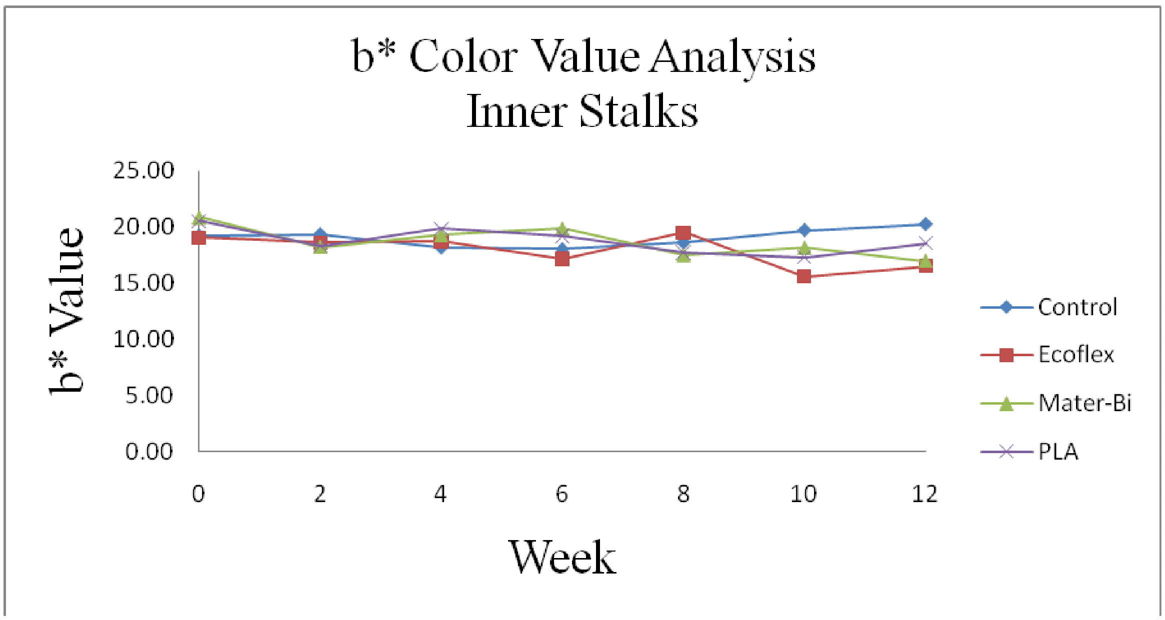

Figure 5.3: b* Color Analysis: Inner Stalks

The b* color analysis for inner stalks follow a different trend than outer and middle stalks but from a practical viewpoint, the b* values don't change greatly over time. From weeks 8-12, b* value began to level off in contrast to slightly declining as noticed with outer and middle stalks. The outer and middle stalks changed more rapidly to a lighter hue than inner stalks because they experienced greater exposure to ambient conditions and therefore deteriorate earlier in color.

Summary: Color Analysis

L* values for outer, inner and middle stalks had increases in L* following the same trend (with regard to no significant difference between materials) through week 6. At week 6, there were significant differences between materials for inner stalks (celery in control was significantly lighter than celery in Mater-Bi). Nevertheless, these differences were likely due to normal variation between celery sticks because there was no discernable trend. At week 8 there was a significant difference between materials for outer stalks as well as week 10 for inner stalks. There were no significant differences in L* value between materials per week for middle stalks.

Similarly for outer and middle stalks, there were no significant differences between materials for a* value measurement. Inner stalks, which are particularly less green than outer and middle stalks demonstrated significant differences in a* value measurement at weeks 2, 6, 10 and 12.

For b* color measurements, there were significant differences at week 6 and 8 for outer stalks. At weeks 6 and 8, yellowing was significantly less for celery packaged in PLA compared to celery packaged in the other bio-based materials. However, for the middle and inner stalks, there were significant differences between materials at week 4 for middle stalks (celery in Mater-Bi was significantly lighter than others) and at week 6, 10, and 12 for inner stalks. Inner stalks appear less green in color (compared to outer and middle) which predisposes them to greater variability in color measurements. These differences are probably due to random variation in celery product.

Table 5.4: Textural Measurement: Firmness vs. Time

Material	week 0	week 2	week 4	week 6	week 8	week 10	week 12
Control	2.83 a	2.68 a	2.38 a	2.50 a	1.97 b	2.20 a	2.42 a
Ecoflex	2.50 a	2.52 a	2.24 a	2.74 a	2.58 a	1.94 a	1.24 b
Mater-Bi	2.3 a	2.72 a	1.88 a	2.38 a	2.64 a	1.97 a	1.94 a,b
PLA	2.65 a	2.28 a	2.62 a	2.53 a	2.68 a	2.78 a	1.15 b

*Within columns, materials with same letter are not significantly different

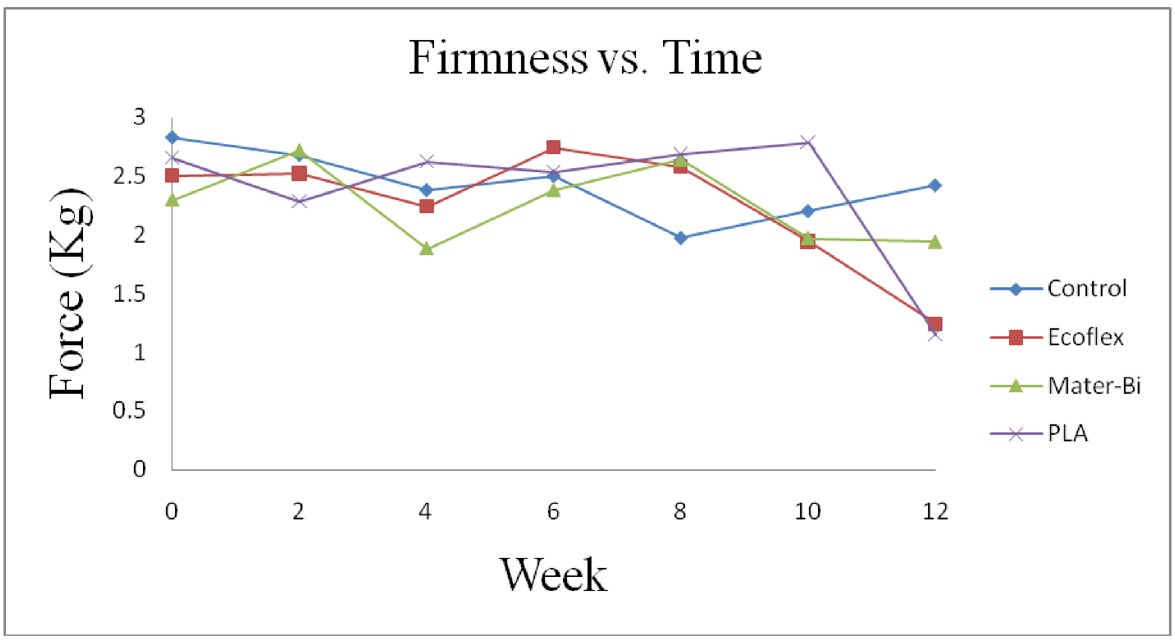

Figure 5.4: Firmness vs. Time

There was large fluctuation in firmness of packaged celery over time. From weeks 0-6, there were no significant differences between materials per week. The only significant difference occurred at week 8, where the celery in the control material was less firm than the others. By week 12, a different trend occurs with celery in control material achieving significant difference in firmness than celery in Ecoflex and PLA.

Table 5.5: Textural Measurement: Stiffness vs. Time

Material	week 0	week 2	week 4	week 6	week 8	week 10	week 12
Control	1.76 a	1.31 a	1.02 a	1.63 a	1.14 b	1.14 a	1.64 a
Ecoflex	1.21 a	1.87 a	1.01 a	1.97 a	1.46 a,b	1.17 a	0.79 b
Mater-Bi	1.3 a	1.60 a	1.12 a	1.45 a	1.7 a,b	1.35 a	1.07 a,b
PLA	1.43 a	1.17 a	1.60 a	1.83 a	1.93 a	1.5 a	0.48 b

*Within columns, materials with same letter are not significantly different

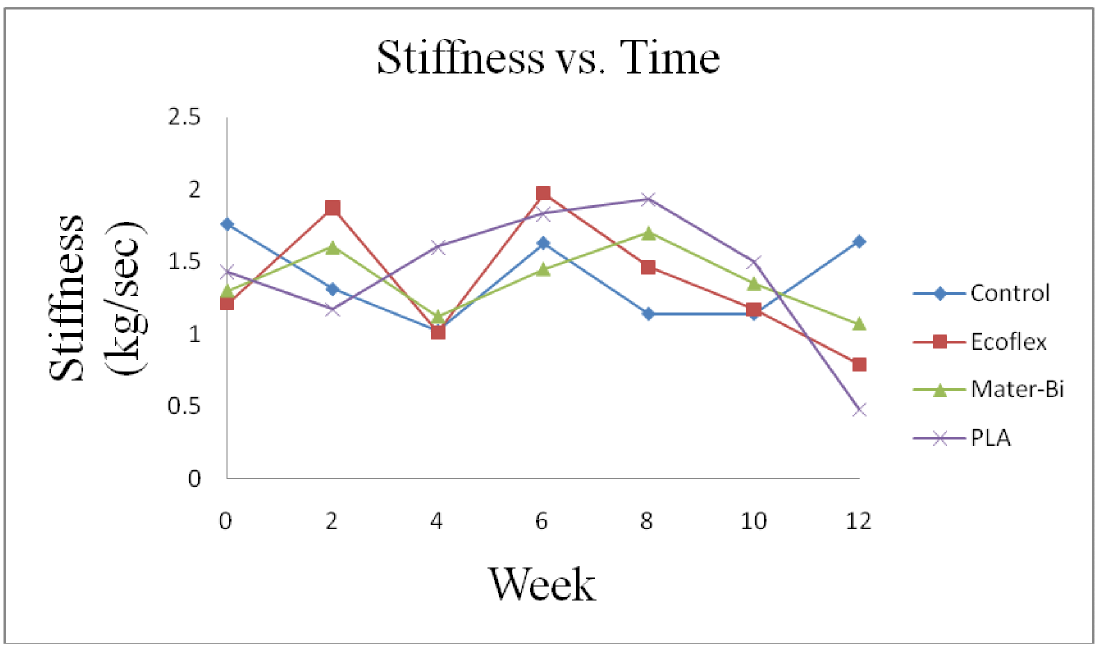

Figure 5.5: Stiffness vs. Time

Although there appears to be large fluctuation in trend, there were no significant differences in textural measurement of celery packaged in each material from week 0-6 and at week 10. Significant differences were noticed in weeks 8 and 12. At week 8, stiffness of celery in PLA was significantly higher than celery in control. However, by week 12, the trend reverses with celery in control significantly higher in stiffness than celery in PLA. Differences in both weeks are possibly due to inherent product variation.

Table 5.6: Textural Measurement: Toughness vs. Time

Material	week 0	week 2	week 4	week 6	week 8	week 10	week 12
Control	7.14 a	6.97 a,b	5.76 a	6.28 a	5.88 b	6.50 a	6.9 a
Ecoflex	6.24 a	7.68 a	5.60 a	7.14 a	7.33 a,b	5.66 a	3.6 b
Mater-Bi	6.78 a	6.9 a,b	4.0 a	7.10 a	7.6 a,b	6.41 a	6.12 a,b
PLA	6.10 a	5.72 b	5.94 a	6.67 a	8.0 a	7.20 a	3.24 b

*Within columns, materials with same letter are not significantly different

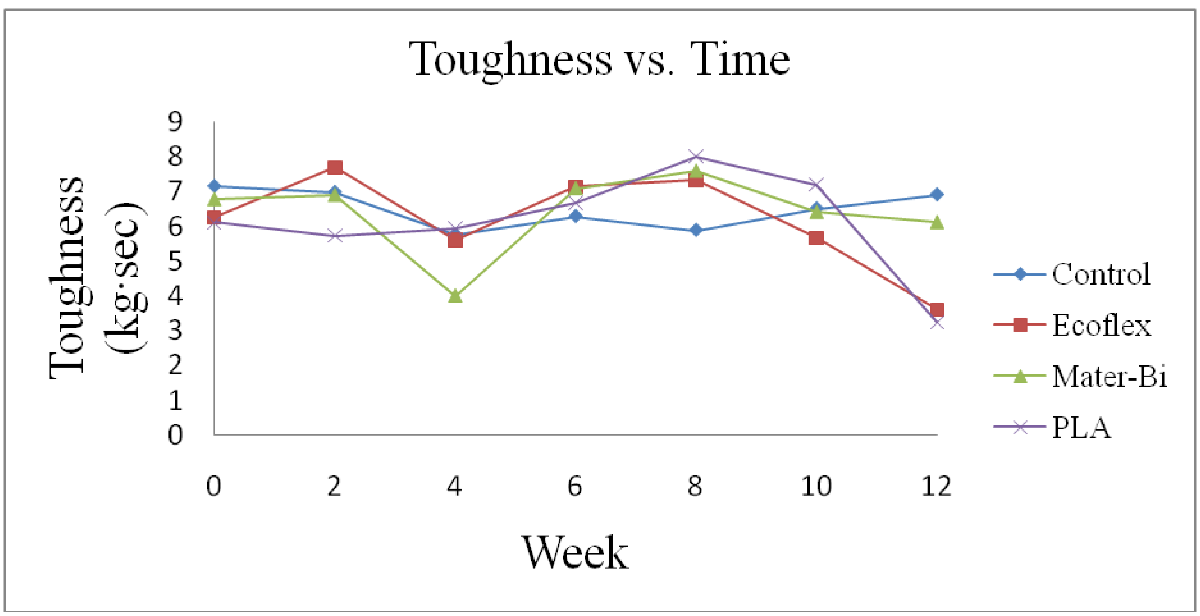

Figure 5.6: Toughness vs. Time

While toughness of celery in all materials except PLA seemed to fluctuate, the toughness of celery in PLA material appeared to be constantly increasing from week 2-8 and declined thereafter. Along with firmness, textural quality of celery includes toughness and rigidity. While there appeared to be some variation in trend over time, there were also significant differences between materials at weeks 2, 8, and 12. Celery in Ecoflex is significantly tougher than celery in PLA at week 2 while at week 8, celery in PLA is significantly tougher than the control. At week 12, celery packaged in both Ecoflex and PLA show decreases while Control and Mater-bi level off. Celery in control film also appears to be significantly tougher than celery packaged in Ecoflex and PLA at week 12.

The variation in textural measurements between materials over time is probably due to variability in product. Similarity in textural variability trends can also be identified in previous research involving shelf life of celery. Variation in texture along with fluctuation in textural measurements was noticed by Vina and Chaves (2003) in a study investigating textural changes in fresh cut celery during refrigerated storage. The shear force required to cut celery sticks increased during the first two week from an initial value of 40.5 N to 47.8 N at day 14. After day 14, the shear force required to cut the pieces decreased to 43 N due to softening of tissue. Further firmness measurements using a texture analyzer also generated trends that depicted variation in measurements over time as studied by Rizzo and Muratore, 2008. Rizzo and Muratore noticed that petioles softened and firmness decreased during storage in all materials. Additionally, no significant differences were noticed during the shelf life period (one month) between both films (polyolefin anti-fog (AF) and polypropylene micro perforated (MP) materials) of packaged celery.

Total Aerobic Plate Count

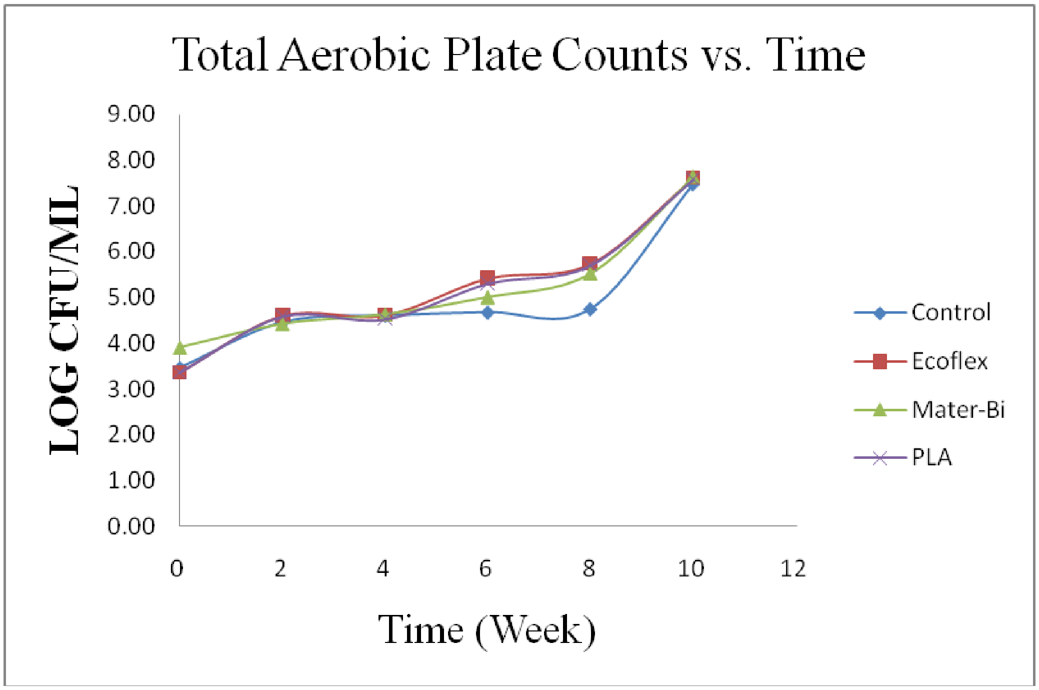

Figure 5.7: Total Aerobic Plate Counts vs. Time

Microbial growth of celery increased during storage in all materials. The microbial growth for celery in the control material was lower by about 1-2 log from weeks 6-8 but eventually reached 7 log CFU/g along with other materials by week 10. 7 Log CFU/g had been identified by Gomez et al as the maximum aerobic bacterial limit for celery.

Table 5.7: Sensory Analysis

Aroma	0=lack aroma	15=aromatic				
Aroma	Material	week 0	week 2	week 4	week 6	week 8
	Control	9.30 a	6.63 a	7.42 a,b	6.61 a,b	7.7 a
	Ecoflex	15.0 a	8.82 a	8.6 a	7.24 a,b	8.72 a
	Mater-Bi	9.13 a	7.12 a	5.4 b	8.52 a	7.6 a
	PLA	7.19 a	8.73 a	8.11 a	5.75 b	7.46 a
Appearance	0=old	15=fresh				
Appearance	Control	11.10 a	6.77 b	9.39 a	7.47 b	7.49 a,b
	Ecoflex	9.89 b	11.09 a	9.98 a	7.57 b	7.61 a,b
	Mater-Bi	10.41 a,b	10.56 a	9.22 a	9.11 a	7.23 b
	PLA	11.03 a,b	11.11 a	9.18 a	7.73 a,b	9.43 a
Flavor	0=bitter	15=sweet				
Flavor	Control	9.59 a	6.85 a	7.95 a	6.81 a	6.16 b
	Ecoflex	8.23 a	8.86 a	8.84 a	8.51 a	6.73 a,b
	Mater-Bi	9.18 a	8.13 a	7.75 a	7.25 a	8.31 a
	PLA	8.43 a	8.03 a	8.61 a	8.01 a	7.95 a,b
Like of Flav.	0=dislike	15=like				
Liking of flavor	Control	11.03 a	6.59 a	9.03 a,b	7.25 a	6.70 a
	Ecoflex	9.78 a	10.3 b	10.00 a	8.67 a	7.17 a
	Mater-Bi	9.92 a	9.04 b	8.08 b	8.83 a	8.57 a
	PLA	9.66 a	9.14 b	9.11 a,b	7.81 a	8.38 a
Texture	0=rubbery	15=crisp				
Texture	Control	11.59 a	9.43 b	10.65 a	9.31 a	7.68 b
	Ecoflex	10.45 a	12.25 a	10.67 a	9.88 a	6.99 b
	Mater-Bi	11.57 a	11.60 a	8.89 b	10.49 a	8.36 a,b
	PLA	11.35 a	11.66 a	10.47 a	9.46 a	10.2 a
Overall Lik.	0=dislike	15=like				
Overall	Control	10.90 a	6.63 b	9.35 a,b	7.84 a	6.97 a
	Ecoflex	9.65 a	10.77 a	10.13 a	8.37 a	7.47 a
	Mater-Bi	10.57 a	9.38 a	7.87 b	9.42 a	7.86 a
	PLA	9.95 a	10.05 a	9.25 a,b	8.07 a	9.13 a
Rank	1=like most	4=like least				
Rank	Control	2.14 a	3.4 a	2.08 b,c	2.67 a	3.22 a
	Ecoflex	2.43 a	1.93 b	1.84 c	2.53 a	3.11 a
	Mater-Bi	2.78 a	2.53 b	3.23 a	2.07 a	2.00 b
	PLA	2.64 a	2.13 b	2.85 a,b	2.73 a	1.67 b

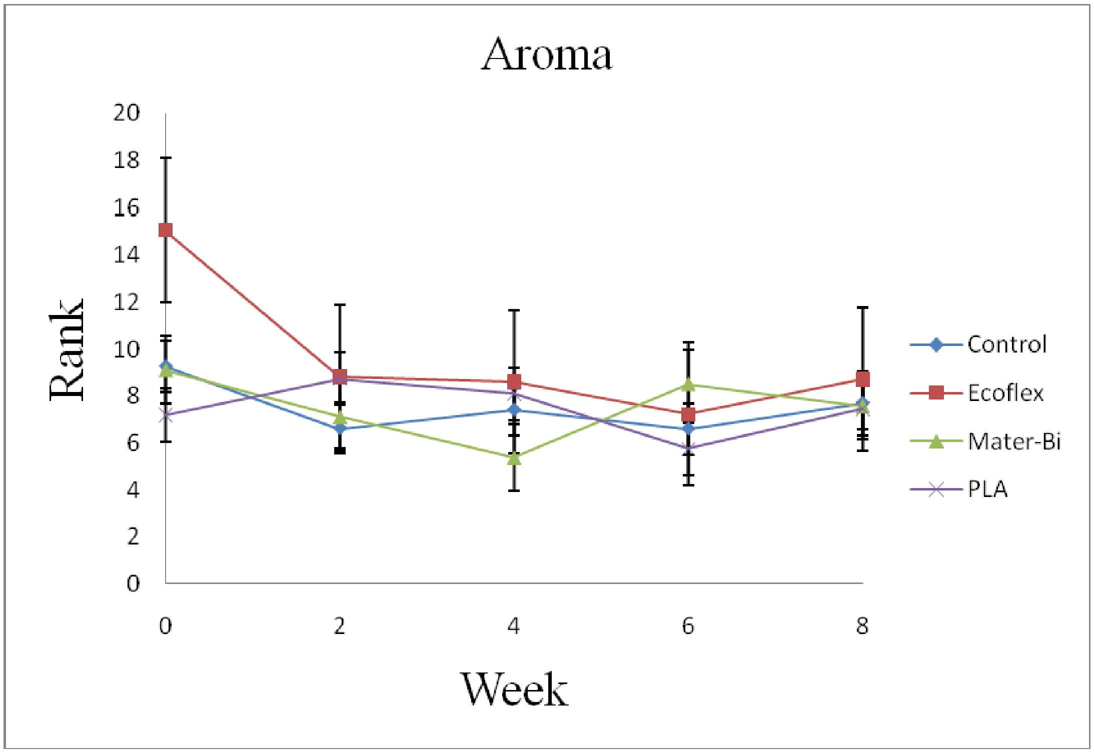

Figure 5.8: Sensory. Aroma Profile

At week 4, celery stored in Mater-Bi was scored significantly less than Ecoflex and PLA but reversed trends by week 6. By week 6, celery in Mater-Bi was scored significantly higher than PLA while others showed no significant differences. During weeks 0,2, and 8, there were no significant differences between materials with regard to aroma. Therefore, the differences observed for celery in Mater-Bi were likely due to normal variability found in produce rather than actual differences. Essentially, aroma was not affected by material differences.

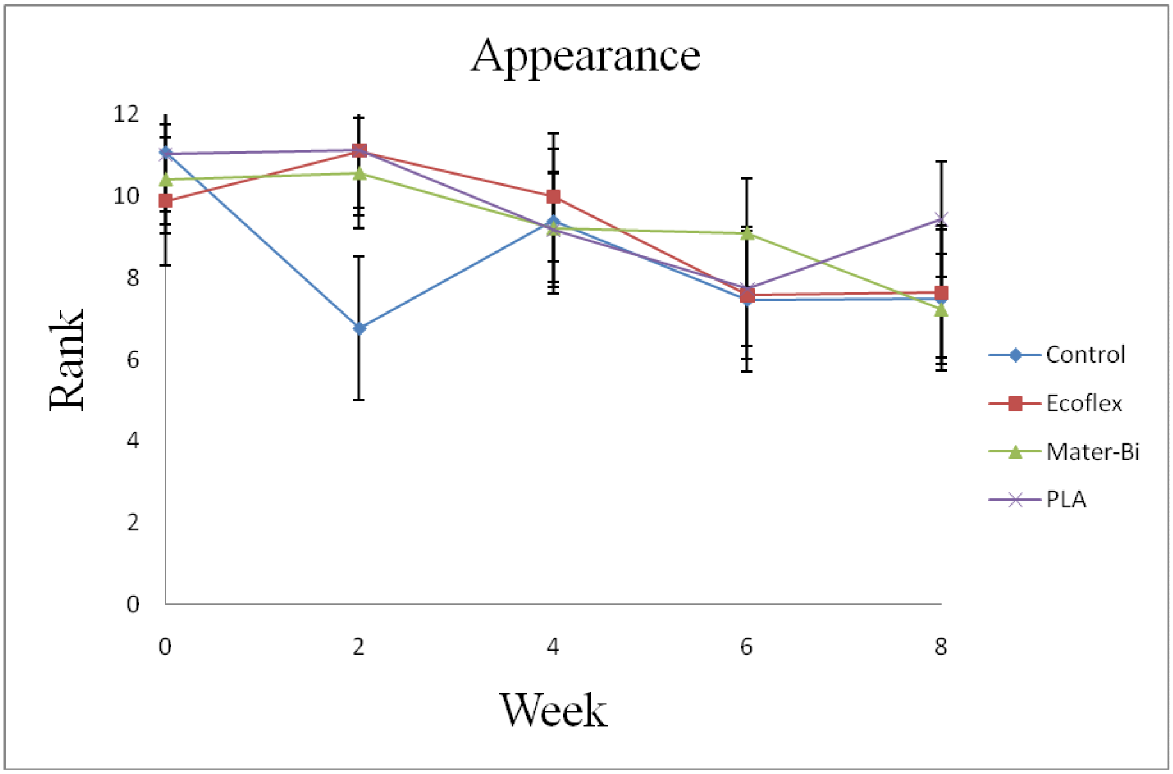

Figure 5.9: Sensory. Appearance Profile

There was a great deal of variation with regard to appearance. The only time a set of samples was ranked below 7.5 (which was the point at which samples fall closer to the unacceptable part of the sensory scale), was for the control at week 2. There was no diserable trend throughout the 8 week evaluation period. Based on statistical analysis, the celery in control film was ranked significantly lower in appearance at week 2, celery in Mater-Bi was ranked significantly higher in appearance than Control and Ecoflex at week 6 and

PLA was ranked significantly higher in appearance than Mater-Bi at week 8. In general, the variation could again be related to normal variability within and between samples and statistical differences are not likely due to diffrences between materials.

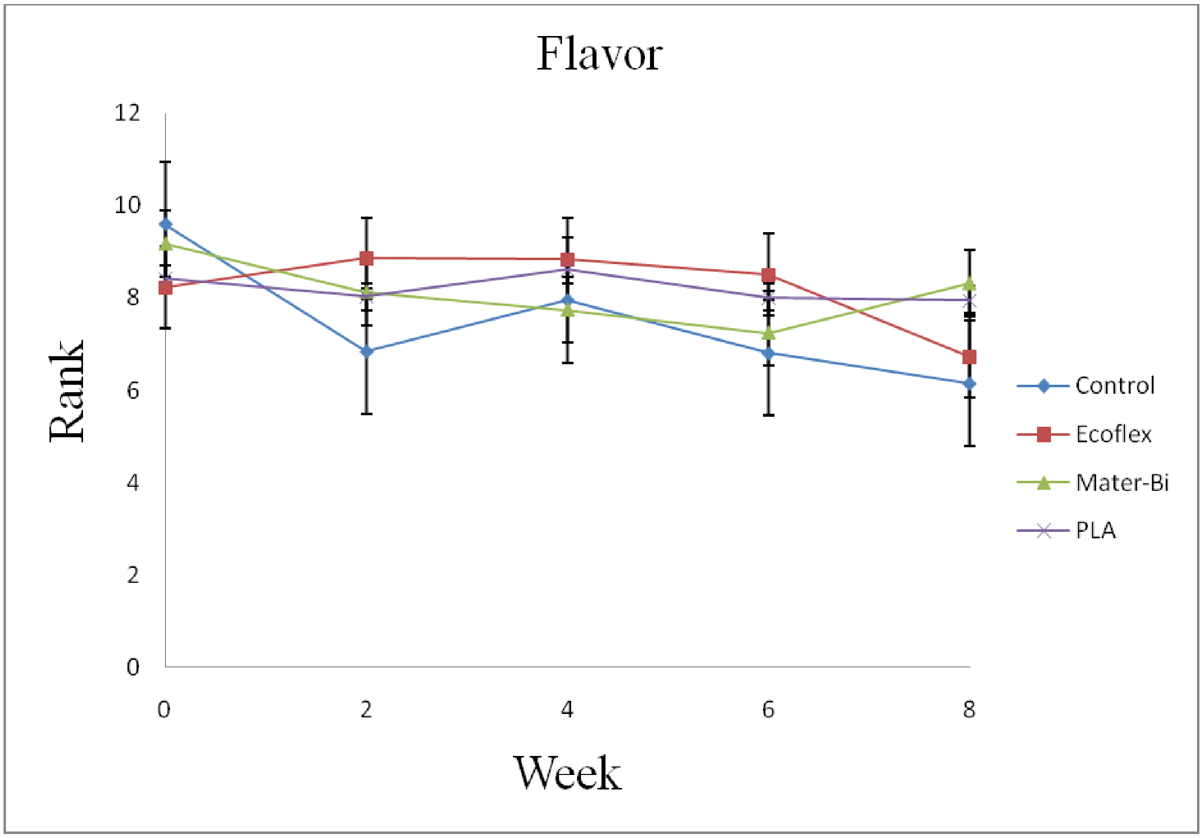

Figure 6.0: Sensory. Flavor Profile

Flavor for all materials was ranked lower at week 8 compared to week 0 but did not change dranstically. There were no significant differences between celery in all materials from weeks 0-6 but at week 8 celery in Materi-Bi was ranked signficantly higher in flavor than celery in other materials.

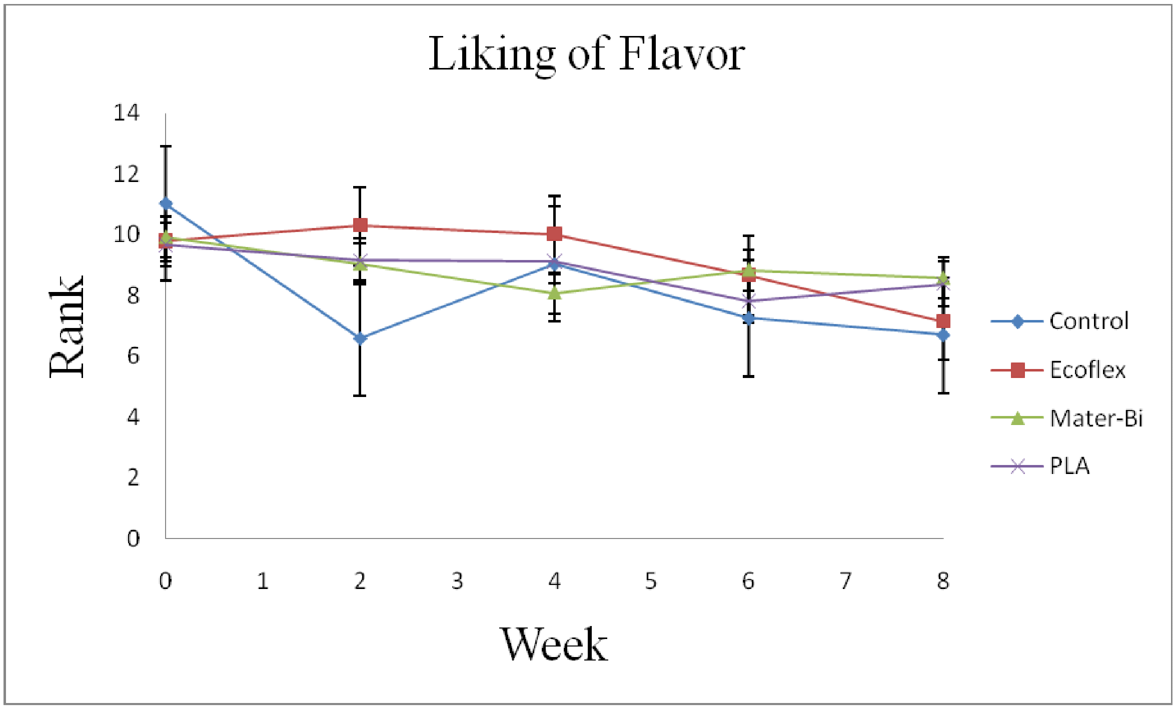

Figure 7.0: Sensory. Liking of Flavor Profile

Generally, the liking of flavor category seem to decline from week 4-8

There is a similarity in trend between "flavor" and "liking of flavor categories" which indicates that panelists utilized their perception of 'flavor' in assessing their perception for 'liking of flavor. At week 2, celery in the control film was ranked significantly lower than rest. Similarly, in the flavor profile above, the control sample was also ranked lower than the rest at week 2.

At week 2, there was a significant difference in the "Liking of Flavor" category.

Generally, the "liking of flavor" category seem to decline from week 4-8

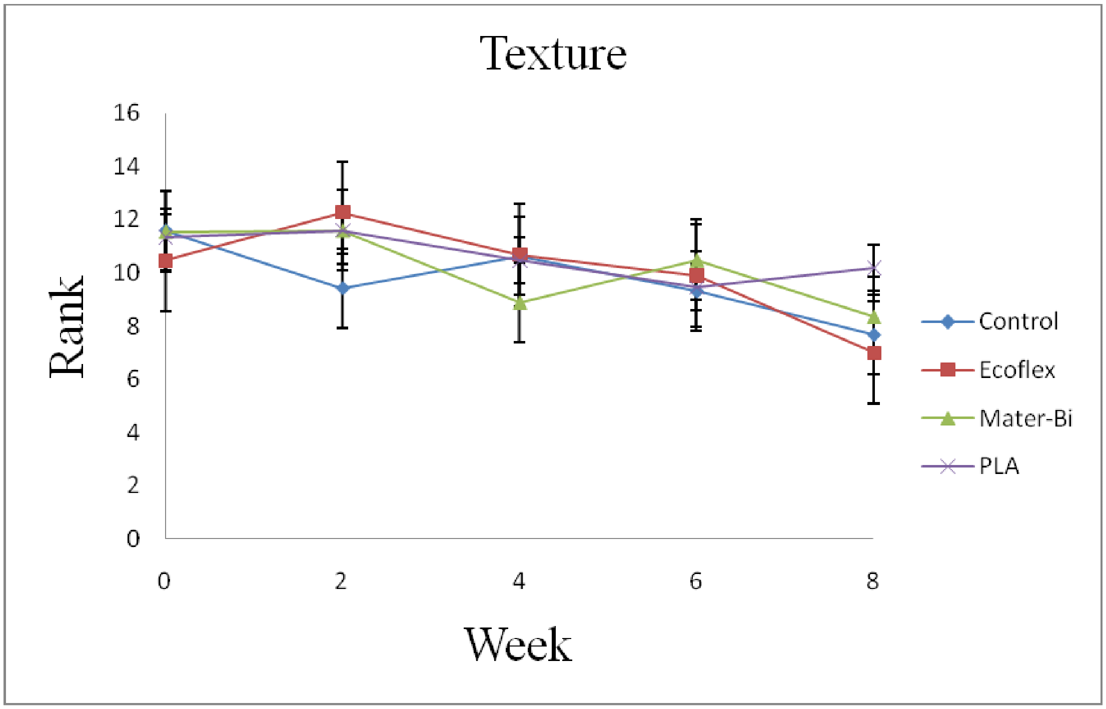

Figure 7.1: Sensory. Texture

Based on the sensory evaluation of texture over the 8 week period, texture declined for all samples. Again, some statistical differences were observed but no specific trend was established. Celery in the control material was scored significantly lower (least crisp) than others at week 2 while celery packaged in Mater-Bi was scored significantly lower than others at week 4. At week 8, celery in all other materials were scored similarly except PLA which was ranked higher (more crisp).

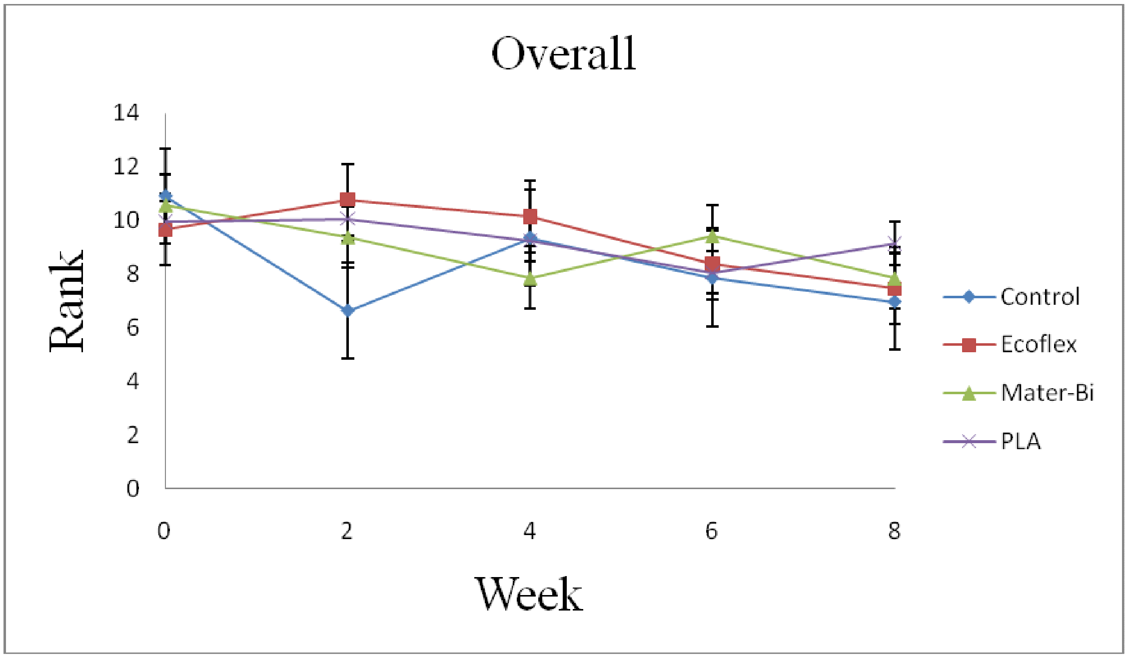

Figure 7.2: Sensory. Overall Profile

The results from the overall category shows a decline from weeks 2-8 with some fluctuation occurring samples in the Mater-Bi and control materials. The results also indicate that there is a similarity between the texture trend and the overall trend. At week 2, celery in the control material was ranked significantly ranked lower than other samples. Similarly, control was also ranked significantly lower for texture at week 2. At week 4, the Mater-Bi sample was also ranked significantly lower than all other materials which compared to results observed for texture when compared to celery in Ecoflex alone. . The indication of same samples ranked significantly different from others for two sensory attributes reveals that both attributes are relevant in assessing each other and panelists utilize both in assessing quality of celery samples.

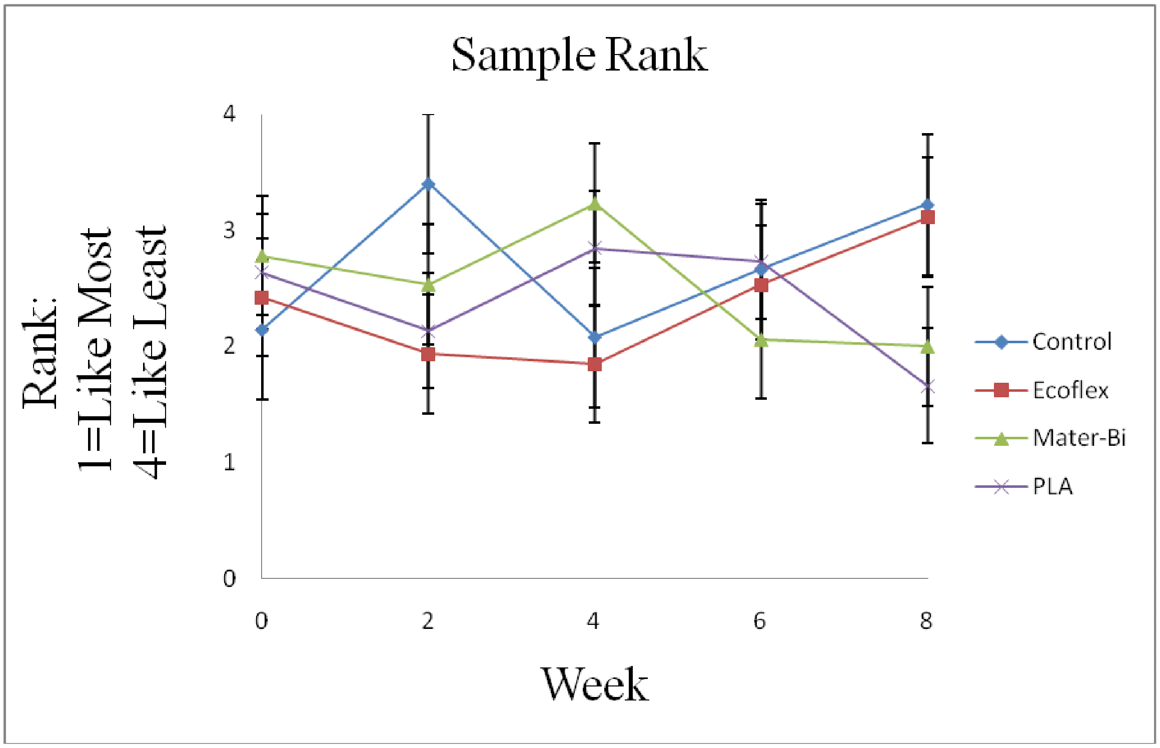

Figure 7.3: Sensory. Ranking Profile

Results of ranking indicate that there were no significant differences between samples at weeks 0 and 6. The greatest variation between sample ranking occurred at weeks 2 and 4 where the celery in control film was ranked least liked ($p<0.05$) and then rankings changed drastically at the next sampling period. At week 4, celery in Ecoflex was ranked the most liked ($p<0.05$) and celery in Mater-Bi was ranked least liked ($p<0.05$). At the end of the sensory testing, the samples broke into to two distinct rankings. The celery in the two biopolymer films (Mater-Bi and PLA) were ranked most liked and the celery in the control and Ecoflex were ranked lower with regard to liking ($p<0.05$).

Summary of Sensory Results

- Aroma: some variation but no significant differences
- Appearance: Celery in PLA was scored more fresh than others
- Flavor: Celery in Mater-Bi and PLA ranked significantly higher
- Liking of flavor: No significant differences
- Texture: Celery in PLA was ranked significantly higher than celery in control and Ecoflex but not significantly different than celery in Mater-Bi
- Overall: There are no significant differences
- Ranking: Celery in Mater-Bi and PLA were most liked

Flavor and other relevant quality attributes analyzed via sensory evaluation in combination with aerobic microbiological analysis are critical in determining end of shelf life. In a shelf life study involving diced celery, Prakash and colleagues (2000) linked aerobic bacterial load surpassing 8 Log to an end of shelf life. Furthermore, edible quality based on aroma, color, and texture was deemed inedible and unacceptable at time period (day 19) of study when microbiological levels surpassed 8 Log. A comparable correlation was also established to current work that links increasing microbiological enumeration to increasing overall dislike of samples and flavor over time. As microbiological analysis of samples increased within each week, overall liking of samples as well as liking of flavor decreased. The proliferation of bacteria appears to impart an influence on perception of sensory attributes which are critical determinants of edible quality and hence shelf life of product. Consequently, the correlation between increasing microbiological enumeration, deteriorating edible quality attributes, and an end of shelf life is established.

MATERIAL ANALYSIS

Table 5.8: Permeation Studies

Table 5.8A: Oxygen Permeability Rate: cc·mil/ (m^2·day) 23°C and 0%RH, 754.17 mmHg

Week	Control	Ecoflex	Mater-Bi	PLA
0	9500	2793	2673	494
6	9508	8181	2850	12,760
12	5990	***	3130	***

Table 5.8 B: Water Vapor Permeability Rate (g·mil/(m^2·day) 37°C and 100%RH,754.17 mmHg

Week	Control	Ecoflex	Mater-Bi	PLA
0	24	995	647	211
6	26	2164	548	225
12	22.09	***	560	***

** indicates extreme variability in results – unable to report accurate values

Much of the variability in data occurred between Ecoflex (OPR and WVPR) and PLA (OPR) materials. Their respective permeability rates had increased drastically by week 12 and became so variable that the permeation equipment was unable to complete the test without failing. Upon observation of the film, it was likely that the film cracked during testing even though the film installed was intact and masked with aluminum foil to reduce the test size area.

At week 12, the control film experienced variability in results resulting in transmission rates higher than weeks 0 and 6. Therefore, the OPR appeared different at week 12 than at weeks 0 and 6. LDPE is a synthetic hydrophobic polymer and should not show permeation decreases impacted by high RH conditions nor display any fluctuations in oxygen permeability data over time. Consequently, the data presented in the above permeation studies may be flawed due to testing error and should be subjected to further analysis.

Mechanical Analysis

Mechanical Analysis

Table 5.9: Break Strength (MPa):

Material	week 0	week 2	week 4	week 6	week 8	week 10	week12
Control	18.36 b	18.0 b	28.1 b	21.61 a,b	21.95 b	23.0 a	19.34 b
Ecoflex	21.75 b	16.1 b	26 b	16.12 b	14.30 c	12.54 b	-
Mater-Bi	29.12 b	35.8 a,b	37.2 b	27.48 a	28.87 a	27.40 a	24.64 a
PLA	50.38 a	50.7 a	66.8 a	*	*	*	*

a,b,c - materials with same letter within columns are not significantly different

* No data recorded. Undetected reading

- insufficient material available (due to deterioration) to conduct testing

There were significant differences in break strength between materials within each week. No tensile strength was recorded for PLA from weeks 6-12 because the film failed immediately upon testing and was probably below the sensing level of the load cell. PLA was the strongest material, indicated by higher values, and also most brittle from weeks 0-4. At weeks 8 and 12, there were significant differences in tensile strength between the remaining materials. Mater-Bi had a significantly greater tensile strength at weeks 8 and 12 while tensile strength for Ecoflex was significantly lowest at weeks 8 and 10.

Break strength is the force required to rupture a film (Instron: Materials Testing). The PLA film required the greatest force to break because it was a brittle material containing "cold" polymer chains. This occurs when the polymer is below its glass transition temperature (T_g). These chains can not easily move or change position when subjected to stress. The inability to move or change positions when subjected to stress prevents them from absorbing any stress placed on them. Consequently, they can either resist the stress imposed on them or break immediately upon sensing stress, as noticed with PLA. Conversely, other materials utilized in the study were ductile materials above their T_g. These polymer chains can change positions easily, because molecules have more kinetic energy required for movement which allows them to easily absorb stress placed on them. A ductile film absorbs more energy before breaking, but the force required to break the film will be lower than a brittle film.

Table 6.0:Yield Stress (MPa):

Material	week 0	week 2	week 4	week 6	week 8	week 10	week12
Control	10.1 b	12.03 b	12.03 b	9.75 b	9.94 b	10.34 b	9.08 b
Ecoflex	*	*	*	*	*	*	*
Mater-Bi	17.29 a	21.05 a	22.06 a	16.44 a	16.77 a	16.65 a	16.80 a
PLA	*	*	*	*	*	*	*

a,b - materials with same letter, within columns are not significantly different

*displayed brittle curves. No data obtained

Yield stress was reported only for control and Mater-Bi films because they displayed ductile curves. PLA displayed brittle curves, therefore no data was reported.

Mater-Bi had significantly higher yield stress than control per week throughout the entire duration of study. Ecoflex is not a brittle material, but its yield point was not sufficiently pronounced to be detected by the software.

The response to force acted on films is identified as yield stress (Instron: Materials Testing). Both ductile materials (Control and Mater-Bi) in Table 6.0 display a yield region under tensile deformation (Figure 7.5 & 7.7). The other two materials do not display yield.

Table 6.1: Break Elongation (%):

Material	week 0	week 2	week 4	week 6	week 8	week 10	week12
Control	516.30 a	477.93 a	692.63 a	566.57 a	573.7 a	562.10 a	526.77 a
Ecoflex	97.83 b	40.07 c	111.83 c	53.50 c	62.33 c	53.40 c	*
Mater-Bi	330.13 a	331.17 b	342.77 b	289.9 b	303.0 b	298.0 b	251.33 b
PLA	4.43 b	4.87 c	4.20 d	-	-	-	-

a,b,c,d- materials with same letter, within columns are not significantly different

- No data recorded. Undetected reading

* Insufficient material available (due to deterioration) to conduct testing

PLA remained lowest in % break elongation from week 0-4. At week 4, PLA was significantly lower than other materials in break elongation. From week 4, materials were significantly different from each other in break elongation. Beginning at week 6, the control material was significantly greater in break elongation while Ecoflex was the lowest. At week 12, the Ecoflex material severely deteriorated and there was not enough film to conduct a tensile test.

Break elongation is defined as elongation of the specimen to the point where it breaks (break point) (Instron: Materials Testing, 2009). Yield elongation can be recognized as the farthest a material can be stretched without undergoing permanent deformation (Selke, S. E., Cutler, J. D., & Hernandez, R. J. (2004). Both LDPE and Mater-Bi materials displayed large elongation because they are ductile materials. The other two materials also displayed elongation. Figure 7.6 displays a brittle PLA material with minimal elongation (and no necking) compared to Ecoflex. Figure 7.6 displays a PLA curve simply increasing and decreasing with no yield region. Conversely, Figure 7.4 shows a ductile Ecoflex with larger elongation than PLA but no necking. Necking refers to the tendency of a material's width to decrease when stretched (Selke, Cutler, & Hernandez, 2004).

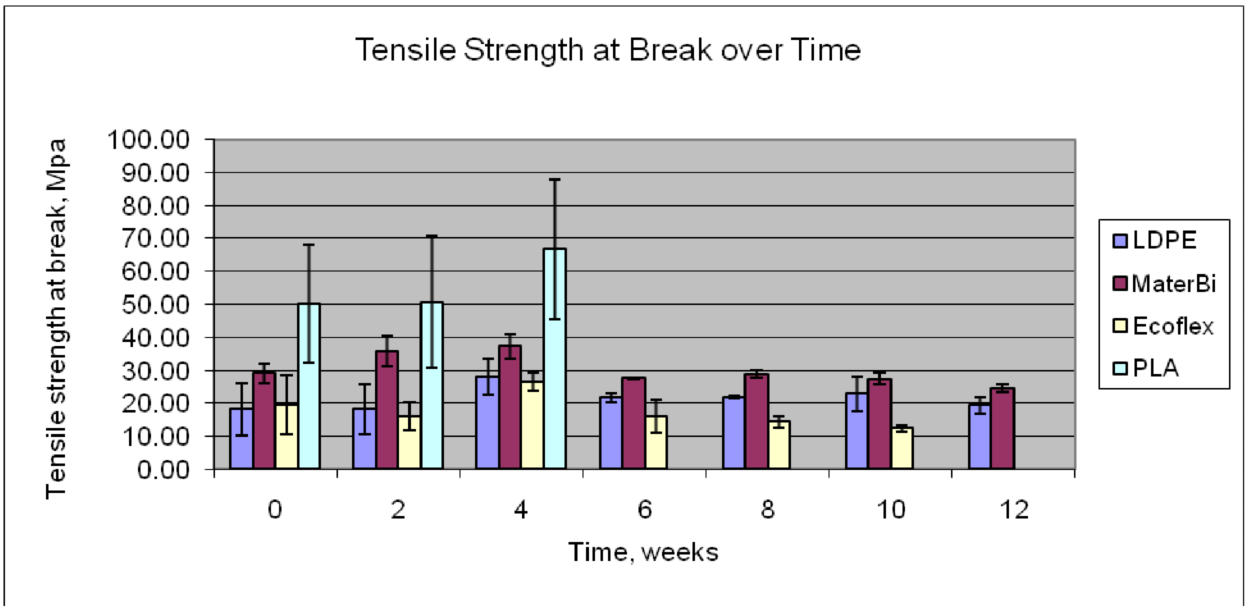

Figure 7.4: Tensile Strength at Break

There is fairly large variability in tensile strength during weeks 2 & 4 for all materials. After week 4, the variability begin to level off from weeks 6-12 for all materials. Mater-Bi demonstrated greater tensile and yield strength than both LDPE and Ecoflex. For instance, at weeks 0 and 2, Mater-Bi had a yield stress of 17.3 Mpa and 21.05 Mpa respectively, while LDPE had a yield stress of 10.1 Mpa and 12.03 Mpa respectively.

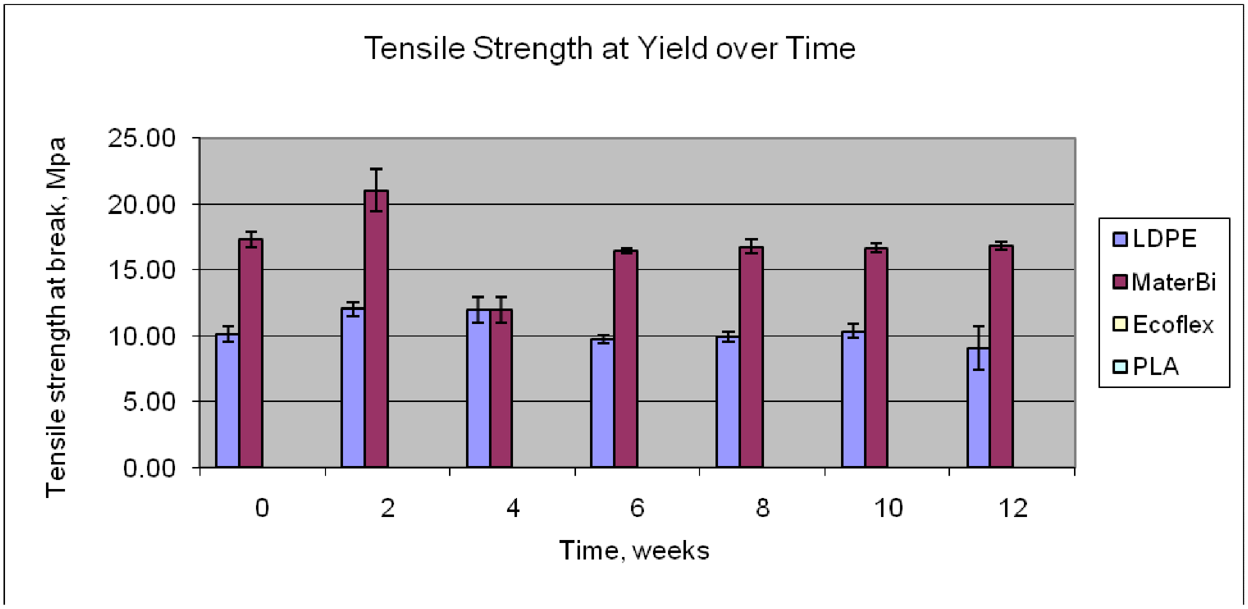

Figure 7.5: Tensile Strength at Yield

Only LDPE and Mater-Bi displayed ductile curves and a detectable yield stress point on the stress strain curve. Ecoflex was also ductile but displayed no yield point. The yield stress between materials within each week were significantly different from each other.

Table 6.2: Curve Shape: Week 0-12

Material	Shape
Control	ductile
Ecoflex	ductile with no necking
Mater-Bi	ductile
PLA	brittle

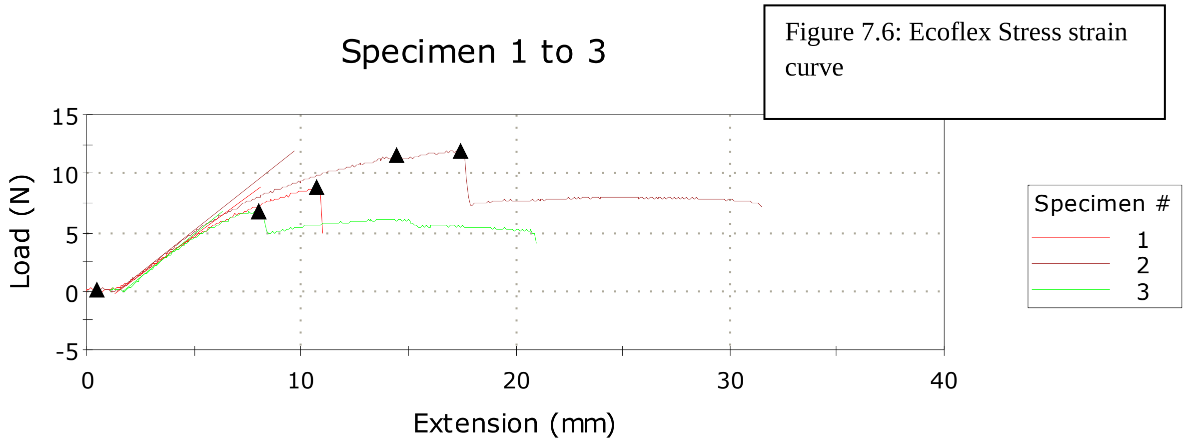

Figure 7.6: Ecoflex Stress strain curve

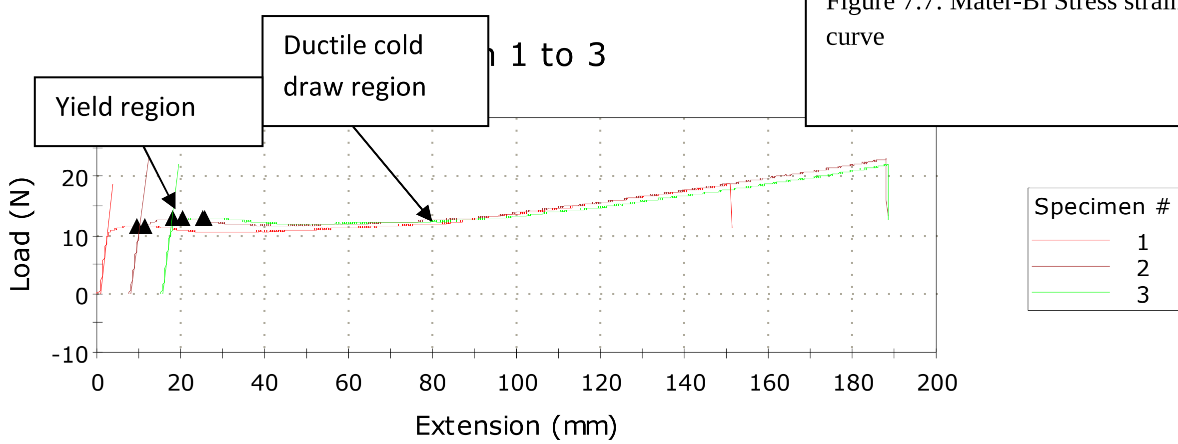

Figure 7.7: Mater-Bi Stress strain curve

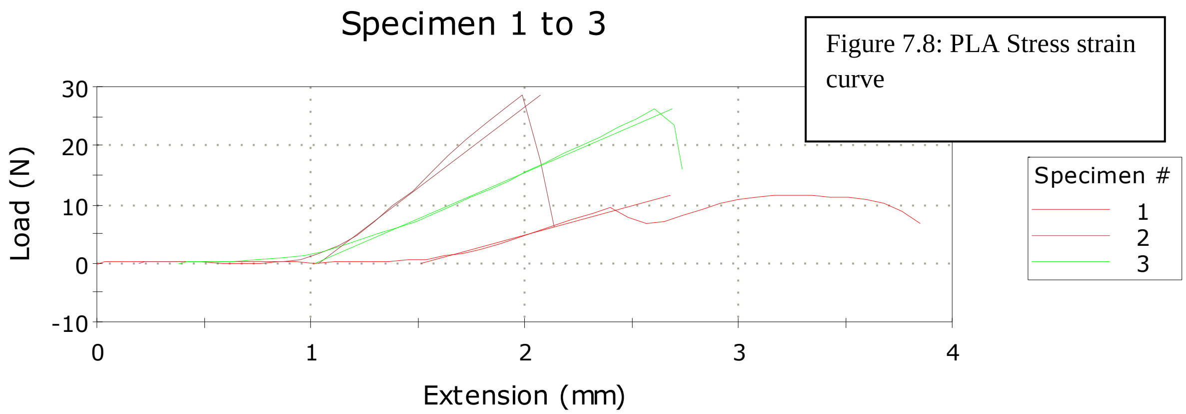

Figure 7.8: PLA Stress strain curve

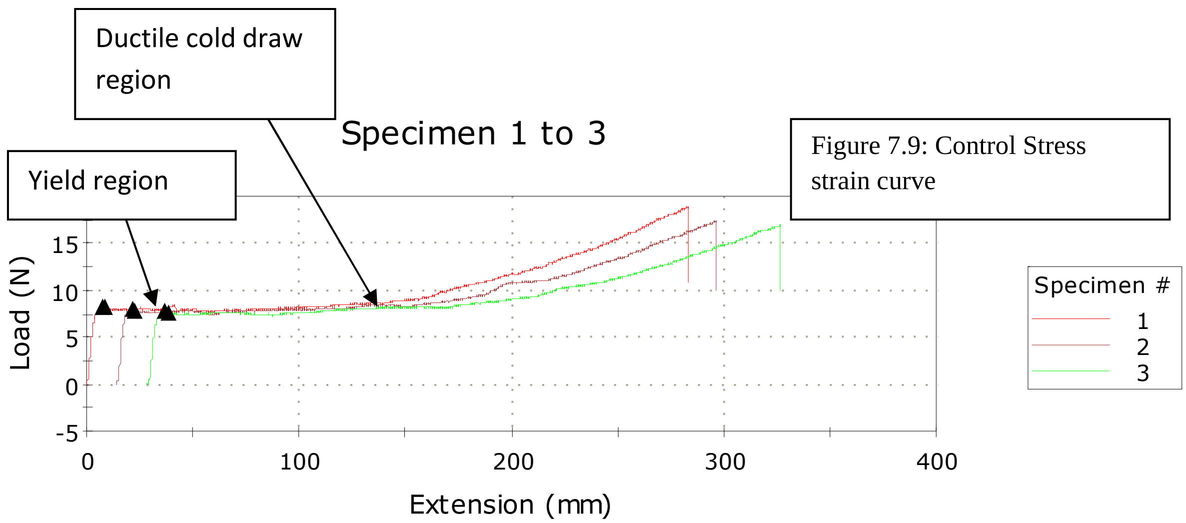

Figure 7.9: Control Stress strain curve

The mechanical behavior of materials used in this study were evaluated by their stress strain characteristics under tensile deformation. Stress is measured in force per unit area and expressed in Pascal or psi. Each material behavior is influenced by rate of deformation. Both the Control and Mater-Bi materials exhibit elastic elongation as indicated by their yield point and ductile cold draw regions. Ecoflex demonstrated no real yield point, and an immediate elongation. Conversely, PLA material displayed an inability to endure a significant amount of strain before breaking because polymer chains were below the glass transition temperature. The glass transition temperature (T_g) is the point where the change in mobility takes place. Below T_g, polymer chains are rigid and exhibit little movement. Consequently, when force is acted on them, they are incapable of absorbing the force, thereby breaking upon impact. On the other hand, control, Ecoflex and Mater-Bi materials contain polymer chains above T_g. Their polymer chains display mobility required to absorb force placed on them. With regard to the main mechanical functions of a package (contain, protect, and utility of use), the Mater-Bi material performed best of the bio-based materials used in this study.

Appearance of Materials:

* view appendix for pictures of material deterioration

Both Ecoflex and PLA materials deteriorated at end of study due to poor mechanical performance under high humidity conditions

The Mater-Bi material remained intact and showed no deterioration

DISCUSSION

Materials utilized in this shelf life study were selected based on their variations in water vapor permeability rates in addition to being sustainable hydrophilic biopolymers derived from renewable resources. A high moisture product (celery) was utilized in studying the effect of these materials on quality and shelf life of product. In analyzing the effect of materials on quality of celery, various measurements were conducted which include texture, color, weight measurements (moisture loss), and sensory analysis. In addition, permeation and mechanical analysis were also conducted on materials over time in order to relate potential changes in material properties to the product quality changes.

Some quality measurements conducted to assess celery quality over time indicated no significant differences between materials within a given week. For instance, texture measurements indicated no significant differences between materials per week. There were differences between weeks which indicated deteriorating quality over time as a function of time but not between materials. Additionally, inherent variation in celery quality was noticed due to random differences between certain weeks that display no particular trend. Such differences exist due to inherent variability in celery product.

Another quality attribute, color analysis, displayed no significant difference in ΔE for overall degree of color change. However, there were some significant differences within materials in the individual color scale measurements. Celery in the Ecoflex material was found to be significantly lighter than celery in other materials in an overall assessment throughout entire study. Likewise, there were also significant differences within weeks that reveal natural deterioration of product over time. The color scale a* is particularly relevant for a green celery product in determining loss of greenness over time. Celery packaged in all materials lost greenness overtime. The b* color scale measurement indicated that celery in Mater-Bi material exhibited significantly higher yellowing while other materials were not significantly different from each other.

Moisture loss was experienced by celery in all materials over the course of 12 week study. However, celery in the Mater-Bi material experienced the least moisture loss over the course of the study and was therefore significantly different than the control material. Overall, celery in the Control material lost significantly more weight than Mater-Bi and PLA but was statistically similar to Ecoflex. There was no significant difference in overall % moisture loss between Ecoflex, Mater-Bi, and PLA materials. Throughout the entire study, celery stored in the Mater-Bi material lost the least amount of weight at 9.3%.

Correspondingly, the Mater-Bi material also showed less variability in WVPR compared to the Ecoflex material which was significantly greater in celery weight loss (moisture loss) in comparison to celery in the Mater-Bi material at end of study.

Sensory analysis revealed significant differences within materials per week. At week 8, celery in Mater-Bi material was ranked significantly higher (more desirable) than celery in control film on the basis of flavor. Similarly, at week 2, celery in PLA film was ranked significantly higher (more crisp) in texture profile than celery in the Control film. At week 8, PLA was also ranked significantly higher than Control and Ecoflex films on the basis of texture. Celery in PLA and Mater-Bi films were liked the most at week 8 and significantly differed from others in a final sensory ranking of samples. In general, sensory analyses indicate that there are some significant differences in certain attributes such as texture and flavor within weeks that are in favor of the variable materials. In attributes with no significant differences within weeks, the variable materials strongly compete with or match the control.

Sensory testing results indicate that some of the variable materials can maintain quality attributes just as the control material. Where there are no significant differences between materials, quality attributes are the same irrespective of material utilized in packaging celery. Consequently, celery could be packaged in any of the materials utilized in the above study and maintain natural quality attributes over time as well as naturally deteriorate over time.

While the celery product can naturally degrade overtime, the materials used in packaging could also deteriorate as noticed with the Ecoflex and PLA materials. Both materials also displayed the highest increases and fluctuation in permeation rates. Additionally, their mechanical performance was also poor compared to Mater-Bi and Control materials. While Ecoflex and PLA materials were suitable materials with respect to product quality attributes, their mechanical properties could be improved to match the performance of Control and Mater-Bi materials.

CHAPTER 5

SUMMARY

The quality attributes evaluated in this shelf life research study were moisture loss, color analysis, texture analysis, and sensory analysis. Additionally, permeation and tensile tests were conducted to evaluate permeability and mechanical properties. All attributes in variable materials were evaluated and compared to each other. Although the control material was also used for comparison, direct correlations cannot be made since it had perforations and the bio-based materials did not. The main purpose of the control was to use it as benchmark since it is the most commonly used package material used in the fresh whole celery market.

There were significant differences in % weight loss between materials per week. Control material experienced the greatest % weight loss ($p<0.05$) from weeks 6-10 while at end of study, Mater-Bi experienced the least% weight loss. Both Mater-Bi and PLA were significantly different than the control in attaining the least increase in % moisture loss overall.

All materials showed increases in ΔE, indicating that all materials showed an overall change in color. Individual color value measurements demonstrated some differences between materials per week in addition to random variability in celery product over time. Celery in Mater-Bi material showed greater yellowing than the PLA material but was not significantly different from the control. Additionally, the Mater-Bi celery was also significantly less light than the control in overall L* color value analysis for the outer stalks. The b* color value analysis showed no significant differences between the variable materials and the control. Over the entire 12 week period, there were no significant differences in a* color value between variable materials and the control. Celery in all materials demonstrated a loss of greenness over time.

Textural attributes demonstrated severe variability in product in much the same way as sensory analysis. By week 8, there were no significant differences between variable materials and the control. However, for the sensory flavor profile, celery in Mater-Bi material was scored significantly higher than the control by week 8. Additionally, for the texture profile, celery in PLA scored significantly higher (more crisp) than the control by week 8 and was also significantly preferred more than the control (sample rank category) at week 8. Along with PLA, Mater-Bi was also ranked as significantly preferred more than the control at week 8 for the sample rank category of sensory analysis.

Microbial growth increased over time for all materials and reached 6 Log by week 8, after which sensory analysis ceased.

Permeation studies depict Mater-Bi as showing the least fluctuation (of the renewable materials) in WVPR. Mater-Bi also performed best at moisture retention and the texture sensory profile. Alternatively, PLA performed better at greenness retention and experienced the least degree of total color change (ΔE).

Overall, Mater-Bi performed better than the other materials with regard to weight loss, WVPR, flavor, sensory rank, and tensile testing. Based on these results, a renewable biomaterial such as Mater-Bi, could possibly replace the nonrenewable material for fresh celery.

CHAPTER 6

RECOMMENDATIONS

The stress strain test performed in this research demonstrated degradation for both Ecoflex and PLA materials. Future research can be conducted in order to improve their ability to resist degradation. Both films could be processed to contain different additives that may aid in improving performance. Such additives can include synthetic polymers such as polyethylene-vinyl alcohol blended with starch to offer flexible films with improved mechanical properties and lower water sensitivity (Mariniello, et al., 2007). The Mater-Bi film used in this shelf life study is made of corn extracted starch to which different synthetic polymers were added to increase flexibility and hydrophobicity. Consequently, the Mater-Bi material performed better than Ecoflex and PLA in moisture retention and tensile tests.

Considerable week-to-week variability in conducted measurements was attributed to natural variation within the celery stalks. Studies of this variability would be a desirable precursor to future work, with celery or other similar high moisture content vegetables such as lettuce, cabbage, and asparagus. Additionally, a larger sample size could also be used to possibly offset natural variation of product.

Future work on shelf life study of assorted produce should also utilize perforated bio-based materials along with perforated LDPE or un-perforated LDPE along with un-perforated bio-based materials.

APPENDIX A: Plastic Tensile Testing Data

Film	Week	Break Strength (MPa)	Yield Stress (MPa)	Break Elongation (%)	Curve Shape
Control	0	18.36	9.86	102.2	Ductile
Ecoflex	0	21.75	28.08	97.8	Brittle
Mater-Bi	0	29.12	17.29	330.2	Ductile
PLA	0	50.39	-----	4.4	Brittle

Control	2	18.0	12.03	477.9	Ductile
Ecoflex	2	16.1	-----	40.1	Brittle
Mater-Bi	2	35.8	21.05	331.2	Ductile
PLA	2	50.7	-----	4.9	Brittle

Control	4	28.1	12.03	692.7	Ductile
Ecoflex	4	26		111.8	Brittle
Mater-Bi	4	37.2	22.06	342.8	Ductile
PLA	4	66.8		4.2	Brittle

Control	6	21.61	9.75	566.6	Ductile
Ecoflex	6	16.12	17.45	53.5	Brittle
Mater-Bi	6	27.48	16.44	289.9	Ductile
PLA	6	-----	-----	-----	Brittle

Control	8	21.95	9.94	573.7	Ductile
Ecoflex	8	14.3	17.26	62.3	Brittle
Mater-Bi	8	28.87	16.77	303	Ductile
PLA	8	-----	-----	-----	Brittle

Control	10	23	10.34	562.1	Ductile
Ecoflex	10	12.54	16.4	53.4	Brittle
Mater-Bi	10	27.39	16.64	298	Ductile
PLA	10	-----	-----	-----	Brittle

Control	12	19.34	9.08	526.8	Ductile
Ecoflex	12	-----	-----	-----	Brittle
Mater-Bi	12	24.64	16.8	251.3	Ductile
PLA	12	-----	-----	-----	Brittle

Appendix B: Sensory Ballot Sheet

SENSORY PROFILE OF REFRIGERATED CELERY STICKS

ID No.__________ **DATE: ?-?-09**

Place a hash mark (I) through the line indicating your evaluation of each attribute.
Cleanse your palate with water before you begin and as frequently as necessary during the evaluation session.
Please comment on all attributes.

CODE: xxx

Aroma:

Lacks Aroma Aromatic

Appearance:

Old Fresh

Flavor: Typical celery flavor can be bitter and or sweet

Bitter Sweet

Liking of flavor:

Dislike Like

Texture:

Rubbery Crisp

Overall Liking:

Dislike Like

Comments:

Sample Rank: Using code numbers, please rank samples from 1-4
1=like most
4=like least

____ ____ ____ ____
1 2 3 4

Sensory Profile on Refrigerated Celery 10-20-08/Smart G

Product Information

September 2001

BASF Plastics
key to your success

Ecoflex F BX 7011

Product description

Ecoflex F BX 7011 is a biodegradable, statistical, aliphatic-aromatic Copolyester based on the monomers butanediol, adipic acid and terephthalic acid. Ecoflex has properties similar to PE-LD because of its high molecular weight and its branched molecular structure

Processing

Transparent to translucent, semi-crystalline structure with DSC melting point within the range of PE-LD
High ultimate elongation at break and high failure energy (dart drop)
High, but controllable water vapour transmission rate WVTR
MVR (190 °C, 2,16 kg): 3 – 6 ml/10 min.
Good thermostability up to 230 °C
No predrying of pellets
Good processability on blown film lines
Down gaging to 10 µm possible
Weldable and printable

Applications

Ecoflex F has been developed for the conversion to flexible films using the blown film process. Potential applications are:
Packaging films
Agricultural films
Hygienic films
Compost bags
We supply technical service information concerning the blown film process with Ecoflex F BX 7011 on demand.

Form supplied and storage

Ecoflex F is supplied as lensshaped pellets in 1 t octabins. Temperatures during transportation and storage may not exceed 70 °C.

Food legislation

Ecoflex F BX 7011 fulfils the requirements of DIN V 54900 for compostable and biodegradable Polymers, because it can be degraded by micro-organisms, which are available in compost and soil. Ecoflex is one of the few biodegradable plastics, which basically complies in its composition with the European food stuff legislation for food contact. Specific limitations and more details are given on request. The converter or packer has to check the suitability of the article for the application.

Product safety

During processing of Ecoflex F BX 7011 small quantities of styrene monomer may be released into the atmosphere. At styrene vapour concentrations below 20 ppm no negative effects on health are expected. In our experience, the concentration of styrene does not exceed 1 ppm in well ventilated workplaces - that is were five to eight air changes per hour are made.

Note

The statements in this document are based on our present technical knowledge and experience. They do not relieve processors of the responsibility of carrying out their own tests, and purchasers of our products are expected to carry out receiving inspections. Neither do they imply any binding assurence of suitability for a particular purpose. Any proprietary rights should be respected and existing legislation observed.

BASF Aktiengesellschaft
Business Unit Polystyrene
D-67056 Ludwigshafen

Internet adress: http://www.basf.de

Plastics

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Ecoflex F BX 7011

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Properties of Ecoflex F BX 7011

Property	Test Method	Unit	Value
Mass density	ISO 1183	g/cm^3	*1,25 - 1,27*
Melt flow rate MFR 190 °C, 2,16 kg	ISO 1133	g/10 min	*3,3 – 6,6*
Melt flow rate MVR 190 °C, 2,16 kg	ISO 1133	ml/10 min	*3,0 – 6,0*
Melting point	DSC	°C	*110 - 115*
Shore D hardness	ISO 868	-	*32*
Vicat VST A/50	ISO 306	°C	*80*

Properties of blown film, 50 µm

Property	Test Method	Unit	Value
Transparency	ASTM D 1003	%	*82*
Tensile strength	ISO 527	N/mm^2	*32/36*
Ultimate strength	ISO 527	N/mm^2	*32/36*
Ultimate Elongation	ISO 527	%	*580/820*
Failure Energy (Dyna-Test)	DIN 53373	J/mm	*14,3*
Permeation rates:			
Oxygen	DIN 53380	$ml/(m^2\ d*bar)$	*1600*
Water vapour	DIN 53122	$g/(m^2*d)$	*140*

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D-67056 Ludwigshafen

Plastics

BASF

SAFETY DATA SHEET
Mater-Bi **NF01U**

This Safety Data-Sheet is directed principally to processors, formulators and all users of this substance. The description of physical, chemical and toxicological properties as well as the advise on handling is based on past experience and currently available information. Novamont SpA urges the recipient of this Safety Data-Sheet to read it carefully in order to evaluate hazards, if any, deriving from the use of this substance, and to inform his employers, delegates and customers.
If you have any question regarding the hazards associated with the use of this substance, please contact the Company named in Section 1.

1) IDENTIFICATION OF THE SUBSTANCE/PREPARATION AND OF THE COMPANY

IDENTIFICATION OF THE SUBSTANCE: **NF01U**
CHEMICAL FAMILY: Mater-Bi™ thermoplastic biodegradable polymers.
USE OF THE SUBSTANCE: Production of biodegradable plastics, e.g. bags, shoppers, …
IDENTIFICATION OF THE COMPANY: **Novamont S.p.A**
ADDRESS: Via G.Fauser, 8 I-28100 Novara, Italy
PHONE: +**39.0321.699611** FAX: +39.0321.699601
e-mail: novamont@materbi.com internet: www.materbi.com

2) COMPOSITION/INFORMATION ON INGREDIENTS

CAS NUMBER: N/A
COMPOSITION: Starch, synthetic polyester, plasticizers.
CONCENTRATION: 100%
HAZARDOUS COMPONENTS: none (R-phrases : none, S-phrases : none)

3) HAZARDS IDENTIFICATION

MAIN HAZARD: None that require special labeling.
INGESTION: No current information available.
SKIN CONTACT: No evidence of harmful effects according to currently available information.
EYE CONTACT: No evidence of harmful effects according to currently available information.
SKIN ABSORPTION: No evidence of harmful effects according to currently available information.
INHALATION: No evidence of harmful effects, according to currently available information, as regards the vapor generated at room temperature.

4) FIRST AID MEASURES.

No special emergency measures are necessary for the normal use of the substance.
EYE CONTACT: Flush eyes with clean water for several minutes.
SKIN CONTACT: Wash skin with soap and water to eliminate residuals.
INGESTION: Remove residuals from mouth. If large amounts are swallowed, consult a physician.
INHALATION: Remove from exposure.

5) FIRE-FIGHTING MEASURES.

EXTINGUISHING MEDIA: CO_2, Dry Chem, Foam, Water Spray.
PROTECTIVE EQUIPMENT: Use approved self-contained breathing apparatus.
UNUSUAL FIRE HAZARDS: N/E.
HAZARDOUS COMBUSTION SUBSTANCES: Toxic gases, oxides of carbon.

EXPLOSION HAZARDS: The substance, as shipped, does not pose any dust explosion hazard because it contains essentially no dust particles in size range to support dust explosion. If the substance pellets are ground into powder (of 75μm or less) then the powder would pose dust explosion hazard.

6) ACCIDENTAL RELEASE MEASURES.

No release to air is expected.
ON SOIL: Use normal good housekeeping procedures for clean-up of substance pellets. Provide protection against slipping hazard.
IN WATER: The substance tends to sink. Vacuum up spill pellets and place into drums for disposal.
WASTE DISPOSAL: See Section 13.

7) HANDLING AND STORAGE.

STORAGE: Store in cool, dry, well-ventilated, low fire risk area, away from heat sources. Take precautionary measures against static discharges.
HANDLING: Handle in well-ventilated areas. If dust generation is expected, e.g. during recycling, milling, etc.. use dust respirator.

8) EXPOSURE CONTROLS/PERSONAL PROTECTION.

GENERAL PRECAUTIONS: Good industrial hygiene should be adopted.
VENTILATION: All processing should be carried out in well ventilated areas.
HAND PROTECTION: General purpose gloves. If in molten state, use approved heat resistant gloves.
EYE PROTECTION: Safety glasses.
BODY PROTECTION: Lightweight protective industrial clothing.
RESPIRATORY PROTECTION: If dust generation occur, use approved dust mask. During normal melt processing conditions, vapors of water and glycerol escape from the machinery exit(s); to comply with your local Regulations for exposure limits a local aspiration of vapors may be required.

9) PHYSICAL AND CHEMICAL PROPERTIES.

PHYSICAL STATE: Solid Pellets.
COLOR: Light yellow, opaque.
ODOR: Mild.
BOILING POINT (760MM Hg)°C: N/A.
MELTING POINT °C: 110°C
DENSITY (H_2O=1): 1.3
VAPOR PRESSURE @20°C: N/A.
VAPOR DENSITY (Air=1): N/A.
MOLECULAR WEIGHT: N/E.
% VOLATILES (by volume): N/A.
EVAPORATION RATE (Water=1): N/A.
WATER SOLUBILITY: Only of its plasticizers.

10) STABILITY AND REACTIVITY.

STABILITY: Stable under normal storage conditions.
SUBSTANCES TO AVOID: Oxidizing or reducing agents.
CONDITIONS TO AVOID: Starts to decompose over 200°C.
HAZARDOUS POLYMERIZATION OCCURS: No.

11) **TOXICOLOGICAL INFORMATION.**
According to information available to us the substance is not harmful to health if handled correctly and processed according to the given recommendations.
ACUTE TOXICITY: LD50 not available.
SKIN: No available information.
ORAL: No available information.
EYES: No available information.

12) **ECOLOGICAL INFORMATION.**
The substance is fully biodegradable when introduced into active biological environments (e.g. composting plants). The biodegradation process is affected by temperature, humidity, pH and substratum activity. More detailed information is available from Novamont SpA on request.

13) **DISPOSAL CONSIDERATIONS.**
Dispose of in accordance with local, state and federal regulations.

14) **TRANSPORT INFORMATION.**
SEA TRANSPORT: Substance not subjected to restrictions.
AIR TRANSPORT: Substance not subjected to restrictions.
LAND TRANSPORT: Substance not subjected to restrictions.

15) **REGULATORY INFORMATION.**
LABELING: Substance not subjected to labeling.
SYMBOLS: None
R-PHRASES: None.
S-PHRASES: None.

16) **OTHER INFORMATION.**
The present Safety Data Sheet is written according to the guidelines of European Directives 67/548/EEC & 91/155/EEC and their modifications (93/112/EEC & 2001/58/EEC) and is referred to the pure substance NF01U.
If the substance NF01U is used as a component of another substance/preparation, the present Safety Data Sheet cannot be considered automatically as valid.

Appendix D: Celery Pictures

Week 0 Pictures

Week 12 Pictures

Ecoflex close- up picture: Week 12

PLA close-up picture: Week 12

Mater-Bi close- up picture: Week 12

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